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FUNDAMENTAL EFFECTS OF COLD-WORKING ON CREEP PROPERTIES OF LOW-CARBON N-155 ALLOY

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SUMMARY

Using a combination of microscopic examination, structural analysis by X-ray diffraction, and creep testing, the fundamental effects of cold-working on the creep properties of low-carbon N-155 have been in part established. By cold-rolling is meant rolling at temperatures below that temperature at which recrystallization takes place during the rolling operation.

It has been found that cold-working improved the creep properties through introduction of residual elastic stresses into the lattice, such stresses acting to prevent the movement of the fundamental creep flow units.

In accordance with this finding, an optimum amount of cold-rolling exists for improvement of creep resistance at any given temperature, and amounts of reduction past this critical amount produce an internal configuration which allows internal stress relaxation to occur to a significant extent during the service of the part at elevated temperature. Conversely, for any given amount of cold reduction, such cold reduction can be expected to improve creep resistance only below service temperatures which allow internal stress relaxation to occur in significant amounts during service or test. Further, cold-rolling must be performed at temperatures below a critical value, otherwise significant internal stress relaxation occurs during the rolling operation or the cool down after the rolling operation. It should be noted that this internal stress relaxation, the occurrence of which nullifies the improvement of creep properties by cold-working, is a recovery process occurring at shorter time periods and/or lower temperatures than recrystallization.

Care must be exercised in applying these findings generally. The least that can be said is that they apply to one heat of low-carbon N-155 only. However, by the use of a "standardizing" solution treatment prior to the cold-working operations, the results can in all probability be applied to other commercial heats of low-carbon N-155 bar stock. There is also a growing body of evidence showing that these results are general

for alloys of the N-155 type (see reference 1). However, this must be further verified and the findings are certainly not to be applied to alloys exhibiting pronounced improvements in creep resistance by virtue of precipitation, for example, Inconel X.

INTRODUCTION

This report covers the fourth part of a continuing investigation into the fundamental behavior at high temperatures of austenitic alloys designed for use in aircraft propulsion systems. The first three parts dealt with the fundamental effects of aging on low-carbon N-155 and Inconel X and the effect of chemical composition on high-temperature behavior of solution-treated alloys of the low-carbon N-155 type (references 1 to 4). This report is concerned with the fundamental reasons for improvement in creep resistance of low-carbon N-155 as a result of cold-working. Also, the investigation was carried out to elucidate the conditions under which cold-working can be expected to improve the creep resistance. For purposes of this investigation, cold-working is any working carried out below the temperature at which recrystallization occurs during the rolling operation. This type of recrystallization is said to occur at the "simultaneous recrystallization rolling temperature."

The approach to the problem was twofold. First, the effects of cold-working under various conditions on the creep resistance at 1200° F and on the internal structure of the material were in part determined. For the latter work, optical- and electron-microscopic examination as well as X-ray diffraction patterns were used. Second, the effects of annealing at various times and temperatures on the creep resistance and internal structure of the above cold-worked samples were partially evaluated.

The first step is basic to the purposes of this investigation as the two types of results - creep measurements and structural determinations - were to be correlated. The second step was taken to see if the annealed samples showed some progressive internal structural change which could also be correlated with the changing creep resistance. Such a finding, it was felt, would facilitate solution of the basic problem of why cold-working of low-carbon N-155 alloy improved its creep resistance, that is, the first step outlined above.

This investigation is part of a research program on heat-resistant alloys for aircraft propulsion systems conducted at the Engineering Research Institute of the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

TEST MATERIALS

Low-carbon N-155 alloy in the form of 7/8-inch square bar stock was used in this investigation. It represents part of one ingot of heat A-1726, the entire ingot having been rolled as 7/8-inch square stock. Other portions of this same square stock have been used in the previous investigations of this series (see reference 1).

The chemical composition of the bar stock was:

Chemical composition (percent)

Carbon	0.13	Molybdenum	3.05
Manganese	1.43	Tungsten	1.98
Silicon	0.34	Columbium	0.98
Chromium	20.73	Nitrogen	0.14
Nickel	18.92	Iron	Balance
Cobalt	19.65		

The bar stock was produced from a 13-inch-square billet, being finally rolled to a 7/8-inch round-cornered square bar between 2060° and 1910° F. The details of the processing are given in the appendix.

Prior to use in this particular investigation, the bar stock was solution-treated 1 hour at 2200° F and water-quenched. This procedure was used in order to place the stock in a condition where prior processing had the least possible influence, thus making the results of this investigation applicable to other commercial heats of the same alloy.

EXPERIMENTAL PROCEDURES

The general procedure was first to roll enough solution-treated stock, under the various conditions used, to furnish samples for creep testing in the rolled condition, samples for X-ray and microscopic examination in the rolled condition, and stock for annealing after the cold-rolling. This annealed stock, in turn, was made in sufficient amounts for both creep test samples (for testing in the annealed condition) and for samples for X-ray and microstructural examination. Details of the experimental procedures were as follows.

Cold-Rolling

All rolling was done on a two-high, single-pass, nonreversing mill, with 5-inch rolls. Bars rolled at room temperature were passed through the rolls eight times for each roll setting, rolling alternately on the two pairs of bar faces. Five standard roll settings were used at room temperature to give reductions of approximately 5, 15, 25, 35, and 40 percent in cross section. Because of pronounced work-hardening, these settings were used in succession. Testing was confined to reductions of approximately 5, 15, and 40 percent in this investigation. The sequence of rolling to these reductions is clear from the above. Because of the difficulty of obtaining exactly the desired reductions, some scatter in the reductions used will be noted.

Bars rolled at temperatures above room temperature were first placed in a furnace at a temperature 20° F above the desired rolling temperature. One-half-hour heating times were used as being sufficient for thermal equilibrium to be established between the furnace and the bars. The heated bars were rolled in the same manner as were the bars rolled at room temperature with the exception that only reductions of 15 percent were used for testing purposes and, further, that this reduction was taken in only one roll setting. This latter procedure was used in order to eliminate any reheating and thus any recovery in the reheating furnace which might take place in an accelerated fashion at the elevated temperatures. Samples were prepared with 15-percent reduction at 1000°, 1400°, 1600°, 1800°, 2050°, and 2200° F. All bars were air-cooled after rolling.

Annealing after Cold-Rolling

All annealing was done in small automatically controlled electric muffle furnaces operating in a still air atmosphere. The furnaces were at temperature before placing the samples in them and the annealing period was assumed to start 1/4 hour after placing the samples in the furnace. Annealing temperatures after rolling were standardized at 1200°, 1400°, 1600°, and 1800° F, with times at 1, 10, 100, and 1000 hours being used except for those few cases where a greater number of points on an experimental curve were needed. Four rolled conditions were used in the annealing studies: reductions of approximately 5, 15, and 40 percent at room temperature and 15-percent reduction at 1400° F.

Microscopic Studies

Microscopic studies were carried out as part of the determination of the effects of cold-work on the structure of N-155. The usual metallographic techniques were used in the preparation of samples for optical examination and the preparation of shadow-cast Formvar replicas for use in the electron microscope were carried out as previously described in reference 3.

X-ray Studies

Sample preparation.- Preliminary to any X-ray studies is the preparation of the surfaces from which the diffraction is to take place. Preparation of surfaces free from strains introduced in the preparation process itself was done by electrolytic polishing as previously covered in reference 3.

X-ray diffraction-line width measurements.- Previous work in the field of cold-work has shown that broadening of diffraction lines occurs as a result of cold-working (see reference 5). This has been ascribed to particle size broadening from fragmentation of the grains or a distribution of lattice parameters resulting from residual elastic stresses. It was felt that such effects would be important as far as creep resistance was concerned and accordingly the width of the [220] line of the austenite was measured as a function of the various rolling and heat treatments. Chromium radiation was used to eliminate fluorescent effects and with this radiation the [220] line was at $\theta = 67^{\circ}$, where fair resolution was obtained. The camera was built specifically for the study of this one line and consisted of only one quadrant as shown in figure 1. The specimen was mounted normal to the X-ray beam and rotated slowly about an axis parallel to, but offset from the X-ray beam. This latter construction was occasioned by the desire to cover the largest possible area on the sample surface (cover more grains), and thus obtain diffraction lines with uniform blackening along the diffraction cone.

The films were prepared under controlled conditions of exposure and development. After drying, they were placed in a Leeds & Northrup Knorr-Albers type recording microphotometer and a record was made of the [220] line. Calibration checks showed that the density against exposure characteristics of the film were linear under the conditions used. The half-height width was then taken at the point on the recording at a density halfway between the top and the bottom of the line. Correction for the presence of the α_2 component was done as described previously (see reference 1). Correction for instrumental broadening was done by the method of Warren and by further assuming that the broadness of the [220] line from the sample which was only solution-treated was due entirely to instrumental broadening and by using it as the instrumental broadening factor in Warren's formula (see reference 6). All broadening data reported thus are really relative to the solution-treated sample. The reproducibility of line widths was not better than ±10 percent.

X-ray diffraction-line integrated intensity measurements.- As an aid to determining the basic cause of the line broadening observed, integrated intensity measurements were made on the [111] line of the various cold-rolled and the cold-rolled plus annealed samples. It was here anticipated that extinction[1] effects would be of major interest. Inasmuch as the lowest order line shows maximum intensity variation with variation in amount of extinction, the [111] line was used.

For the majority of work on [111]-line intensities, small samples were cut from the various bars at a plane normal to the rolling direction and the surfaces were prepared with the electrolytic polishing methods. These samples were mounted in a Norelco spectrometer equipped with a copper target, spun in a special fixture previously described (see reference 4), and the [111] lines recorded. As a standard of comparison, a nickel powder compact was used. No theoretical importance is to be attached to the intensity ratios obtained against the nickel standard, however.

In a few special studies it was necessary to determine the variation of [111]-line intensity with variation of the angle between the (111) planes contributing to the intensity measurement and the rolling direction in the bar. For this work, two samples were taken from the bars containing faces cut at 90° and 0° with respect to the rolling direction. These samples were mounted in a Schultz fixture, placed in the Norelco spectrometer, and intensity measurements were taken at 5° or 10° intervals from 90° to 40° and 50° to 0°, respectively, with respect to the rolling direction. The standard of comparison for these intensity measurements was a randomly oriented powder compact made of low-carbon N-155 filings prepared so that extinction was negligible.

Creep Testing

Creep tests were carried out on the various samples with a single stress of 50,000 psi and at the single temperature of 1200° F. The amount of creep testing was limited by the amount of equipment available. Further, the creep tests were run for approximately 200 hours each - long enough to establish the creep rate with reasonable certainty.

These creep tests were run in simple-lever beam-loaded units equipped with automatically controlled electric-resistance furnaces and modified Marten's type extensometers, as previously described in reference 1. It should be emphasized again, however, the creep rates were not reproducible any closer than by a factor of 2.

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[1]Extinction in general is the reduction of the intensity of the diffracted beam due to increasing loss of energy in the primary beam as diffraction itself occurs.

RESULTS AND DISCUSSION

Figures 2 through 9 show the results of investigating the effects of cold-working solution-treated low-carbon N-155 alloy under various conditions on the creep resistance (as represented by the secondary creep rate at 1200° F under a stress of 50,000 psi), internal structure (as represented by diffraction-line widths and intensities), and the microstructure. These results can be divided into two groups. Figures 2 through 5 show the variations in creep resistance and internal structure with amount of reduction at room temperature. Figures 6 through 9 represent the variation with temperature of reduction of the same things plus microstructure, the amount of reduction being held constant at approximately 15 percent.

Figures 10 through 23 show the effect of annealing material rolled under four conditions. Figures 10, 11, and 12 are concerned with creep resistance, [220]-line width, and [111]-line intensity, respectively, of material reduced 5 percent at room temperature and as a function of annealing time with annealing temperature as a parameter. The materials rolled approximately 15 and 40 percent at room temperature and the material rolled 15 percent at 1400° F are covered in order in the three following groups of three figures each. Figures 22 to 24 show representative microstructural changes occurring during annealing. The influence of preferred orientation on the [111]-line intensity is shown by figure 25. Figure 26 is concerned with a correlation of internal structure and creep resistance.

Structural Alterations as a Result of Cold-Working and Cold-Working Plus Annealing

An analysis of the internal structural alterations resulting from cold-work and cold-work plus annealing is given first in order to make the discussion of the effects of cold-work on creep resistance which follows clearer.

Microstructure.- From a microstructural standpoint little change was observed in samples rolled at room temperature until reductions of the order of 15 percent were reached, at which time a few lines could be observed in polished and rather heavily etched samples. Increasing degree of reduction resulted in greater density of such lines at least up to reductions of 40 percent. Figure 5 shows an electron micrograph taken at 10,000X of material reduced 40 percent at room temperature.

Increase in the rolling temperature with the degree of reduction held constant at 15 percent resulted in several microstructural changes. From 80° to 1800° F, the outlines of the grains became clearer, with possibly some precipitate actually formed at the boundaries at the higher temperatures (see fig. 9). The change, compared with material solution-treated only, was similar to that obtained when solution-treated material is heated at the various rolling temperatures for about 1 hour. After rolling at 2200° F, recrystallization was evident (see fig. 9). A check on material rolled at 2050° F revealed no recrystallization (it appeared much the same as material rolled at 1800° F; see fig. 9(c)). The simultaneous recrystallization temperature of low-carbon N-155 for reductions of 15 percent may thus be placed between 2050° and 2200° F. Greater reductions would undoubtedly lower this temperature.

<u>Factors influencing line broadening</u>.- For an interpretation of the widening of the [220] line after rolling (see fig. 3), reference should again be made to the two possible causes of line broadening due to cold-work. They are fragmentation of the matrix into crystallites below about 1000 A or the introduction of a range of interplanar spacings ((110) spacings in this case) in turn due to the presence of residual elastic stresses.

In order to separate these two causes of line broadening use was made of the fact that if the observed broadening were due to crystallites, the crystallites would have to be small enough to eliminate extinction. Accordingly, the first step in the separation of the two causes of line broadening was to make measurements of the [111]-line integrated intensity in order to determine the extinction coefficients[2] in cold-rolled material.

As is evident from figure 4, cold-working increased the intensity of the [111] lines. However, before these intensities could be interpreted in terms of extinction coefficients, a further complication had to be considered. The intensity increase shown in figure 4 could be due not only to reduction in extinction but also to preferred orientation of the crystallites. The latter would act to increase the intensity of various diffraction lines in certain directions relative to the bar axis, such increase not having any relation to extinction decrease.

Figure 25 shows a more detailed study of the intensity of the [111] line at various angles to the rolling direction of materials

[2]The extinction coefficient is here defined as the ratio of the observed [111] integrated intensity to the [111] integrated intensity from a powdered sample of low-carbon N-155 with no extinction.

reduced 15 and 40 percent at 80° F. The curves of figure 25 may be taken as representing the relative number of crystallites having the various indicated orientations with respect to the bar (rolling) axis. Further study showed that the preferred orientation was of the "fibered" type with two components, (111) and (100). The crystallite distribution about any axis parallel with the bar axis was random.

With the results shown in figure 25 in mind, it is obvious that the increase in [111]-line intensity shown in figure 4 was due at least in part to the formation of a preferred structure.

In order to obtain the change in [111]-line intensity due to rolling and corrected for the preferred crystallite orientation, use was made of an important feature of figure 25. With no change in extinction the areas of the curves from 0° to 90° relative to the rolling direction would remain constant to a very close degree, irrespective of whether the fibered orientation is present or not. Thus, any change in this area is due to extinction effects. For the particular curves of figure 25 the height of a rectangle running from 0° to 90° equalling the above-mentioned area would give the [111]-line intensity, relative to the extinctionless low-carbon N-155 powder compact, of a material having the same crystallite size as the rolled bars but with the crystallites completely random.

Construction of such rectangles was carried out in figure 25. For the material rolled 15 percent at 80° F, the resulting [111] intensity relative to the standard was 0.80 and, for the material rolled 40 percent, 1.05 was obtained. The same value for the plain solution-treated low-carbon N-155 was 0.77.[3] The estimated accuracy for all of these figures is ± 5 percent.

Since the standard of comparison for these intensity measurements in figure 25 was powdered low-carbon N-155 with no extinction, the above figures represent extinction coefficients. It is obvious therefore, that rolling 15 percent hardly reduced extinction in low-carbon N-155 over what it was without rolling, while rolling 40 percent eliminated extinction at least to within the experimental error.

If these values of the extinction coefficients are entered into the Darwin formula for extinction as a function of crystallite size, it is found that the as-solution-treated low-carbon N-155 and the stock rolled 15 percent had crystallite sizes of the order of 10^{-4} centimeter, while all that can be said for the stock rolled 40 percent is that its

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[3]Obtained by comparing the [111] intensity of the solution-treated bar stock with the low-carbon N-155 powder compact.

average crystallite size was probably less than 10^{-5} centimeter. Inspection of figure 5 shows that slip lines are spaced less than 10^{-5} centimeter apart and thus some particles of less than 10^{-5} centimeter do exist in this material.

Going back to the original problem of separating particle size broadening from residual strain broadening, the Scherrer formula shows that particle size broadening is negligible with particles greater than 10^{-5} centimeter and so particle size broadening was certainly absent in solution-treated low-carbon N-155 cold-reduced up to 15 percent and of unknown proportions in the material reduced 40 percent. One can thus conclude that with cold reductions of the order of 15 percent, the line broadening which occurred was due entirely to residual elastic strains while the line broadening in samples reduced 40 percent could be due in part to small particle size but not very probably. Accordingly, line widths shown in figures 3, 7, 11, 14, 17, and 20 may be considered as probably indicating some average value of the internal "locked up" stresses after the various treatments indicated.

Recent publications bear upon this matter of particle size broadening as opposed to the residual strain concept. Wood (see reference 7) has shown that under certain rather "refined" conditions of cold-working (tensile pulling) and using methods specifically designed to eliminate the residual elastic stress effects upon the line widths, there is, in truth, a broadening due to particle size. He emphasizes, however, that these results apply only to systems which have undergone relatively homogeneous deformation and the conclusion is that the method of deformation used herein was such as to give a nonhomogeneous system of residual elastic stresses. Further, the rough estimates of Bragg (reference 8) show, by entirely different reasoning than used above, that the particle size broadening of diffraction lines cannot be more than one-half of the total broadening, the balance being due to residual elastic stresses. Lastly, Dehlinger and Kochendoerfer (see reference 9) have found by using precise methods for separation of the two line broadening effects that particle size broadening is negligible when compared with residual stress broadening in severely cold-deformed copper.

Figure 3 indicates that the internal stresses did not increase uniformly with degree of reduction but rather that the internal stresses apparently would reach a maximum value independent of further reduction. These results have been noted before by many investigators.

Figure 7 shows that rolling 15 percent up to temperatures of approximately 1500° F resulted in the same average internal stress. Increasing the temperature of reduction past this point reduced the internal stress and rolling above 2200° F results in practically no residual internal stress. Relaxation occurring during or just after the rolling operation (during cool down) seems to be the controlling factor here.

Figures 11, 14, 17, and 20 indicate that reduction of line width was negligible during annealing at 1200° F for all the materials studied except that reduced 40 percent at room temperature. For the latter material appreciable relaxation occurred at 1200° F. Apparently, the greater the magnitude of the internal stresses is, the lower the temperature at which effective relaxation occurs. This reduction of stability of the cold-worked structure with greater degree of cold-working seems well-substantiated by other investigators of recrystallization phenomena. The two materials reduced 15 percent, one rolled at 1400° F and the other at room temperature, behaved during annealing similarly in regard to changes in line width (compare figs. 14 and 20). The material reduced 5 percent at room temperature exhibited line widths after annealing which were practically independent of time at temperature and a function of temperature of anneal only (see fig. 11). The dependence of line width upon time at temperature appeared to increase roughly with degree of reduction.

Factors influencing diffraction-line intensities.- Study of the line-intensity data in figures 4 and 8 indicates first, that rolling at room temperature in increasing amounts resulted in increasing [111]-line intensities and, second, for a fixed amount of reduction (15 percent) increasing the temperature of reduction had little effect until temperatures around 1600° F were reached. Beyond such temperatures the [111]-line intensity decreased until it approached closely the value obtained from stock solution-treated only.

Figure 25 shows that the major part of this increase in [111]-line intensity in the cold-rolling temperature range was due to the formation of a preferred structure. Further, the results of figure 8 indicate that a preferred orientation was certainly present when rolling was done below about 2000° F. Above this temperature, the presence of a preferred orientation is unlikely.

Figures 12, 15, 18, and 21 indicate that reduction of [111]-line intensity was small during the annealing of the representative rolled materials (reduced 5, 15, and 40 percent at 80° F and 15 percent at 1400° F) except for the material reduced 40 percent at 80° F. This was the only material of the above group which recrystallized under the conditions of annealing (annealing periods up to 1000 hours at 1200°, 1400°, 1600°, and 1800° F).

Figures 22 to 24 show the microstructures of stock reduced 15 percent at 1400° F and stock reduced 40 percent at 80° F that were annealed at 1800° and 1600° F, respectively. Recrystallization was evident in the latter material. The electron micrograph in figure 24 shows that recrystallization of the material reduced 40 percent at 80° F started after about 10 hours at 1600° F. (Note the outlines of a new grain in about the center of the picture.) The sharp reduction in line intensity of this material was a consequence of this recrystallization for the structure after recrystallization was more randomly oriented. Some retention of a preferred orientation after recrystallization of the same general type as existed before recrystallization, although less sharp, has been noted before by other investigators.

The recrystallization of the material rolled 40 percent at 80° F was also evident in another way. The area under the curve in figure 25 for the stock reduced 40 percent was such as to give an average height of 1.05 ± 0.05. After 10 hours at 1600° F, this height was 0.90 and after 100 hours at 1600° F (complete recrystallization) the average height of a curve similar to that of figure 25 was 0.80. This decrease in the extinction coefficient was due to the growth of new larger crystallites and extends a previous observation of Averbach (see reference 10) that reduction in the extinction coefficient occurred early in the recrystallization process in copper filings.

Another rather curious phenomenon was evident during recrystallization of the cold-rolled material. A sharpening of the (111) component of the fibered structure over the (100) component occurred during the annealing periods preceding recrystallization. This shift in crystallite position (possibly a relaxation phenomenon) would explain the actual increase in line intensity, observed in figure 18, which precedes the sharp drops.

Effect of Cold-Working on Creep Resistance

The results of creep testing, under the standardized conditions of 50,000 psi and 1200° F, of the various rolled and annealed samples are shown on figures 2, 6, 10, 13, 16, and 19.

Figure 2 shows a maximum amount of creep resistance from reductions of the order of 25 percent and that further reduction resulted in either no further improvement of creep resistance or possibly an actual reduction. These results probably hold only for temperatures near 1200° F and in all probability would change for other temperatures of creep testing (see reference 11). While this maximum effect of rolling on creep resistance was present, figures 3 and 4 indicate essentially that the internal stresses and the degree of preferred orientation continued to increase with amount of rolling to at least 40-percent reduction.

Figure 6 shows that the temperature of reduction had small effect on creep resistance under the conditions of testing at 1200° F, unless rolling temperatures greater than about 1400° F were used. Above this temperature the creep resistance dropped off sharply. Figure 7 indicates that this drop in creep resistance corresponded closely with the drop in line width. Figure 8 indicates that while a drop in [111]-line integrated intensity occurred with increasing rolling temperature, it occurred at a higher rolling temperature (approximately 1700° F) than did the drop in creep resistance.

Figures 10, 13, 16, and 19 show the relationship between creep resistance and annealing under the standard conditions. All the figures have a common characteristic - increasing time of annealing at either 1200°, 1400°, or 1600° F resulted in a decreasing creep resistance, the rate of decrease being higher, the higher the annealing temperature. When comparison of the creep data obtained from the annealed samples was made with the line-width and line-intensity data obtained from the same samples, two facts were immediately obvious. First, little correlation, if any, existed between creep rate and [111]-line intensity as measured on the bar transverse plane. Second, a correlation between creep rate and line width might exist, but with the creep rate decreasing very rapidly with decreasing line width. Previous studies (see reference 4) of the creep behavior of Inconel X showed experimentally that the logarithmic creep rate under fixed conditions of stress and temperatures was a linear function of the line width (measured as outlined in the section "Experimental Procedures"), the greater the line width the less the logarithmic creep rate. With this in mind, the same correlation of the creep-rate and the line-width data of both rolled and rolled plus annealed samples of this investigation was tried with the result shown in figure 26. Considering the possible errors in measuring creep rates and line widths most points fall no further off the mean line than could be accounted for by experimental error.

Some comment on the significance of this correlation can be made. It is true that if the Eyring reaction rate theory for creep is extended to include some average internal stress, the correlation shown on figure 26 can be predicted. However, the trouble with this procedure is in selecting from line-broadening data an average internal strain and, in turn, converting this to an average internal stress. What the half-height width of a line represents in terms of average internal strain is not known without further analysis of the broadened lines. Even the correction for instrumental broadening using Warren's method is dependent upon an assumption as to the shape of the broadened lines which imperfectly represents the facts in this case. One is forced, therefore, to conclude that the theoretical significance of figure 26 is limited at the present time.

In figure 26, point 3 for the material reduced 40 percent at room temperature falls far off the correlation. The observed creep rate of this material was too high for the measured line width as-rolled. This fact is shown in another way if the data for this material given in figures 2 and 3 are compared (see discussion in the section "Structural Alterations as a Result of Cold-Working and Cold-Working Plus Annealing"). Further, the data of figure 17 showed that the internal stresses of this material (and this material only) relaxed appreciably during annealing at 1200° F. When cognizance is taken of all these facts, the conclusion is that reductions of the order of 40 percent on low-carbon N-155 in the cold-working range resulted in appreciable relaxation of internal stresses at temperatures as low as 1200° F, such relaxation occurring during creep taking place at this temperature. This limits the amount of reduction (and internal stress formation) that is useful for service at 1200° F as shown in figure 2. Further, the line width that should be entered on the correlation of figure 26 is some average line width which existed during the creep test at 1200° F. Obviously, this value is hard to determine. All other samples for which the data lie on the correlation of figure 26 had structures stable enough at 1200° F after rolling and/or annealing that the line widths measured before testing were substantially those existing during the creep test at 1200° F.

The factors explained above which limited the amount of cold reduction that was useful in improving creep resistance at 1200° F to about 40 percent can be generalized somewhat with the aid of data published by Zschokke and Niehus (see reference 11). They also found that there was a maximum benefit from cold-working similar in nature to the results shown in figure 2. Further, the amount of cold-work which gave the maximum effect was found to decrease with increasing temperature. When this result is considered in the light of the structural studies covered herein, it is evident that as a general rule there exists at each temperature of service a critical amount of cold-work beyond which relaxation of the internal stresses will occur to such an extent at the temperature considered that a maximum value for internal stresses effectively exists at each such temperature. Zschokke and Niehus actually showed a reversal in creep strength with increasing amounts of cold-work. Therefore, the relaxation that occurs during service when the critical amount of cold-work is exceeded may be sufficient to more than compensate for any increase in internal stresses initially present over the maximum possible during service. Further, since these internal stresses are the means by which cold-working improves the creep resistance, any additional cold-work past the critical amount is useless. Lastly, the temperature dependence of the amount of cold-work which is critical may be explained by saying that the temperature at which relaxation occurs to any appreciable extent is reduced as the amount of cold-working is increased.

As noted before, the materials reduced 15 percent at either room temperature or 1400° F were similar in every respect in regard to the interrelation between internal structure and creep resistance either before or after annealing. Further, the results shown in figure 6 indicate that internal stresses after reductions of 15 percent are also the same up to temperatures of reduction of the order of 1500° F. Beyond this point relaxation during the rolling operation is occurring. Since it is these internal stresses which improve creep resistance and since removal, through relaxation, of these stresses by annealing after rolling progresses the same over the same temperature range, it can be said that the temperature of cold-work is immaterial as far as creep resistance is concerned over this same temperature range of 80° to 1500° F.

The data for material rolled at 2200° F and the as-solution-treated material also did not fall on the correlation of figure 26 (points 1 and 2). Further, figure 6 showed that within the temperature range of 1800° to 2200° F a rather curious reversal in creep resistance occurred. In view of the above, it seems that the creep resistance for materials rolled in the vicinity of 2200° F and the solution-treated material is abnormally high, if internal stress control as exemplified by figure 26 is considered normal.

No fundamental explanation of this phenomenon appears possible at the present time. However, from a technological standpoint the finding is important since the conclusion is that rolling of solution-treated N-155 at temperatures in the vicinity of 2200° F results in material which has better creep resistance than any other hot-worked material.

One last important observation may be drawn from the data reported herein. After cold-rolling, the temperature dependence of the speed with which X-ray diffraction lines sharpen (which corresponds to the speed with which internal stresses are relaxing) may be used to predict the service temperature range over which cold-rolling will improve creep resistance. Since it has been shown above that the internal stresses existing at any given time strongly determine the creep resistance, it is obvious that cold-rolling will improve creep resistance only if internal relaxation does not occur to any appreciable extent during the expected service life. This may be determined quite easily by simply studying line sharpening (after cold-rolling) as a function of temperature and time. It would further seem that the ability of a material to retain internal stresses after cold-rolling is dependent upon its macroscopic relaxation characteristics in some way. The same mechanisms at internal flow probably control, on the one hand, relaxation on an optical microscopic scale (over distances of the order of 10^{-4} cm) and, on the other hand, relaxation on a macroscopic scale (over distances of the order of several inches). This points to the conclusion that for a material to be improved in creep resistance at some given temperature by cold-working, it must have a good relaxation strength in the ordinary sense of the word in the annealed state at the same temperature. The initial stress for the measurement of ordinary relaxation would have to be the same as some mean internal stress in the cold-worked state.

CONCLUSIONS

From an investigation of the fundamental effects of cold-working on the creep properties of low-carbon N-155 by microscopic examination and structural analysis by X-ray diffraction, the following conclusions may be drawn:

1. Cold-working improved the creep resistance of low-carbon N-155 through the presence of elastic stresses left in the lattice after the working operation. The residual stresses referred to, however, were those which remained during the creep test.

2. A correlation of the logarithmic creep rate against the half-height line width existed for solution-treated and cold-rolled low-carbon N-155 either with or without subsequent anneals. The full theoretical significance of this correlation must await more exact interpretation of broadened diffraction lines.

3. Cold-working to improve creep resistance may be carried out at any temperature below that which will allow relaxation of the internal stresses to occur to a marked degree during the working operation itself. For N-155 alloy, this useful working temperature range includes the so-called "warm-working" range.

4. Cold-worked material may also be expected to show improved creep resistance only below temperatures which allow internal stress relaxation to occur to a marked degree during the expected service life.

5. Conversely, increasing amounts of cold-working give increasing creep resistance only up to the point where the internal stresses are high enough to permit relaxation to occur in significant amount at the particular service temperature being considered. It further appears that the critical amount of cold reduction decreases as the service temperature is raised.

6. It appears that line widths themselves or some other reliable indicator of line widths can be used as a production control of cold-reduced N-155 alloy since the creep rate, encountered in an acceptance test for example, is experimentally a known function of line width.

University of Michigan
Ann Arbor, Mich., June 2, 1950

APPENDIX

PROCESSING OF LOW-CARBON N-155 7/8-INCH BROKEN-CORNER SQUARE BAR STOCK FROM HEAT A-1726

An ingot was hammer cogged and then rolled to bar stock under the following conditions:

(1) Hammer cogged to a 13-inch-square billet
Furnace temperature, 2210° to 2220° F
Three heats - Starting temperature on die, 2050° to 2070° F
Finish temperature on die, 1830° to 1870° F

(2) Hammer cogged to a $10\frac{3}{4}$-inch-square billet
Furnace temperature, 2200° to 2220° F
Three heats - Starting temperature on die, 2050° to 2070° F
Finish temperature on die, 1790 to 1800° F

(3) Hammer cogged to 7-inch-square billet
Furnace temperature, 2200° to 2220° F
Three heats - Starting temperature on die, 2050° to 2070° F
Finish temperature on die, 1790° to 1890° F

Billets ground to remove surface defects

(4) Hammer cogged to 4-inch-square billet
Furnace temperature, 2190° to 2210° F
Three heats - Starting temperature on die, 2040° to 2060° F
Finish temperature on die, 1680° to 1880° F

Billets ground to remove surface defects

(5) Hammer cogged to 2-inch-square billet
Furnace temperature, 2180° to 2210° F
Three heats - Starting temperature on die, 2050° to 2065° F
Finish temperature on die, 1730° to 1870° F

Billets ground to remove surface defects

(6) Rolled from 2-inch-square billet to 7/8-inch broken-corner square bar - one heat
Furnace temperature, 2100° to 2110° F
Bar temperature start of rolling, 2050° to 2060° F
Bar temperature finish of rolling, 1910° F

(7) Bars are numbered 1 through 56; bar 1 represents the extreme bottom of ingot and bar 56 the extreme top position

All billets were kept in number sequence throughout all processing, so that ingot position of any bar can be determined by its number

(8) All bars were cooled on the bed and no anneal or stress relief was applied after rolling

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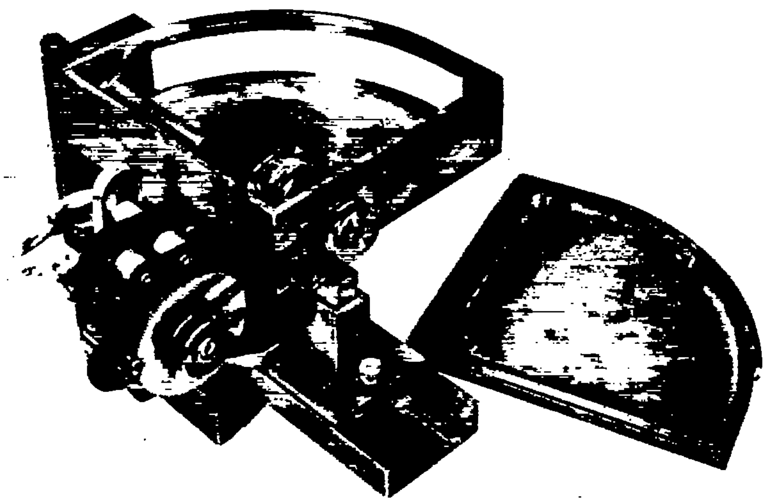


Figure 1.- Back-reflection camera for study of line widths.

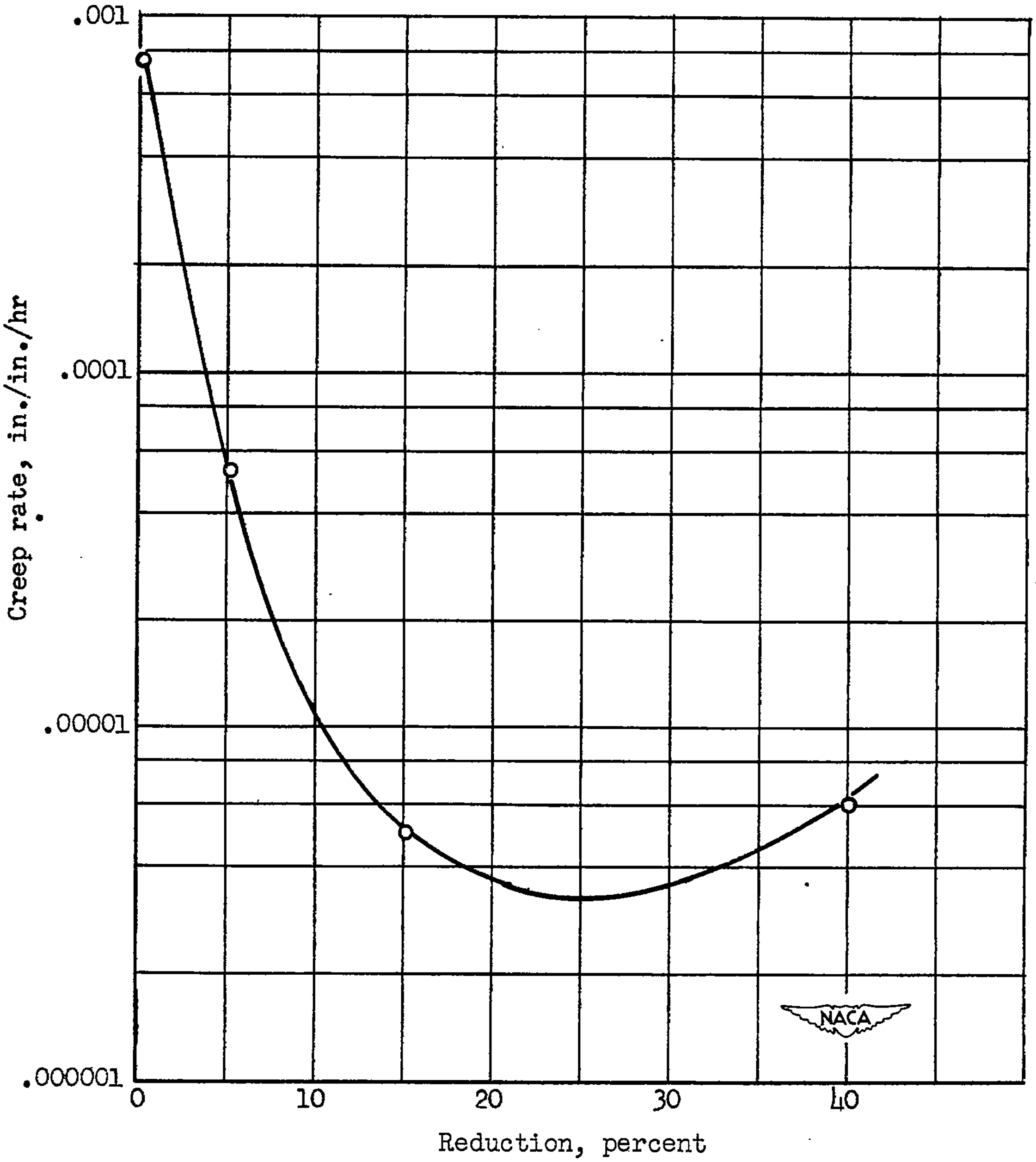


Figure 2.- Effect of reduction at 80° F on secondary creep rates of solution-treated low-carbon N-155 alloy under 50,000 psi at 1200° F.

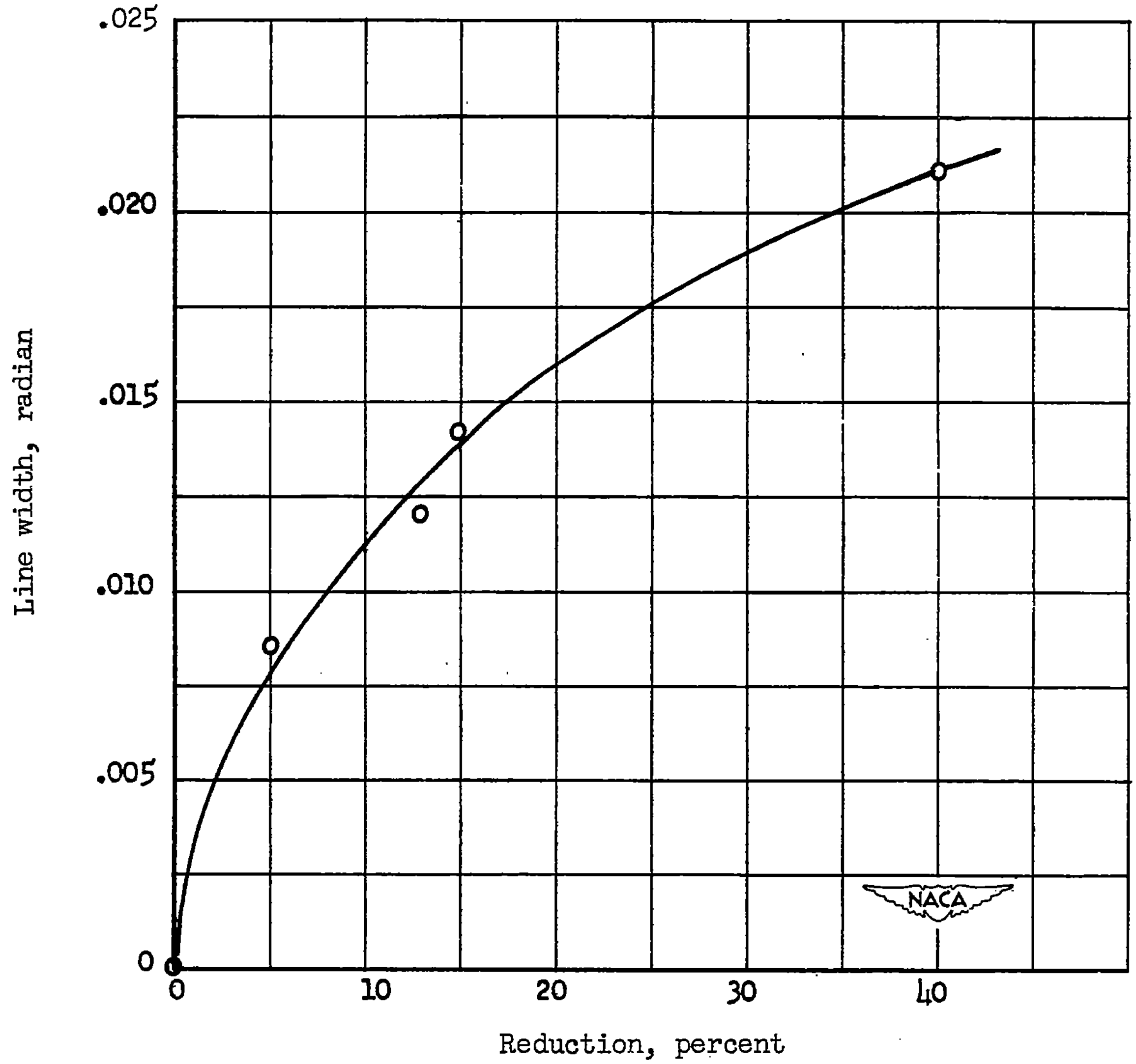


Figure 3.- Effect of reduction at 80° F on width of [220] line of solution-treated low-carbon N-155 alloy.

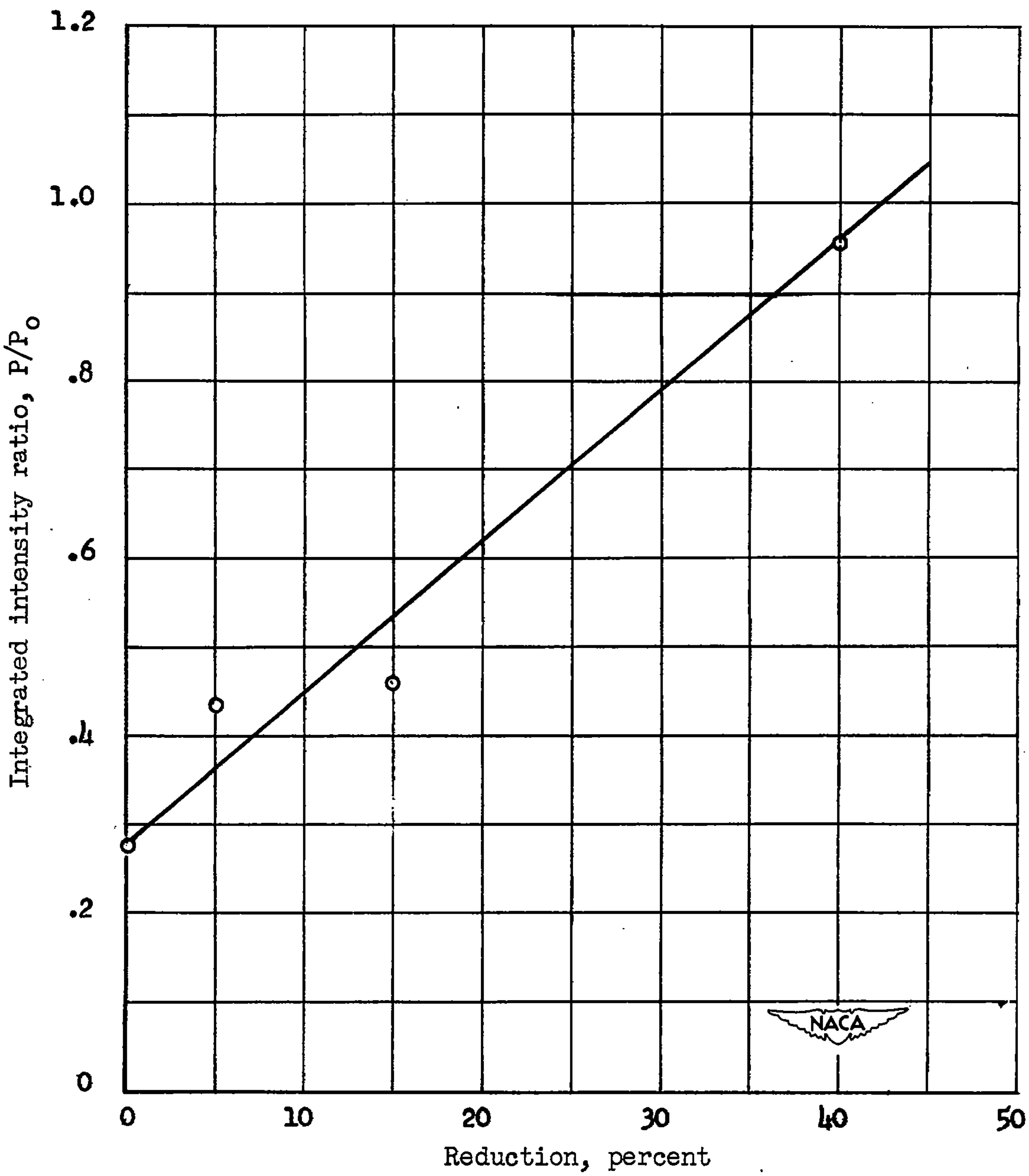


Figure 4.- Effect of reduction at 80° F on intensity of [111] line of solution-treated low-carbon N-155 alloy. P, integrated intensity of [111] line of low-carbon N-155; P_o, integrated intensity of [111] line of nickel standard.

Figure 5.- Electron micrograph of solution-treated low-carbon N-155 bar stock reduced 40 percent at room temperature, X10,000.

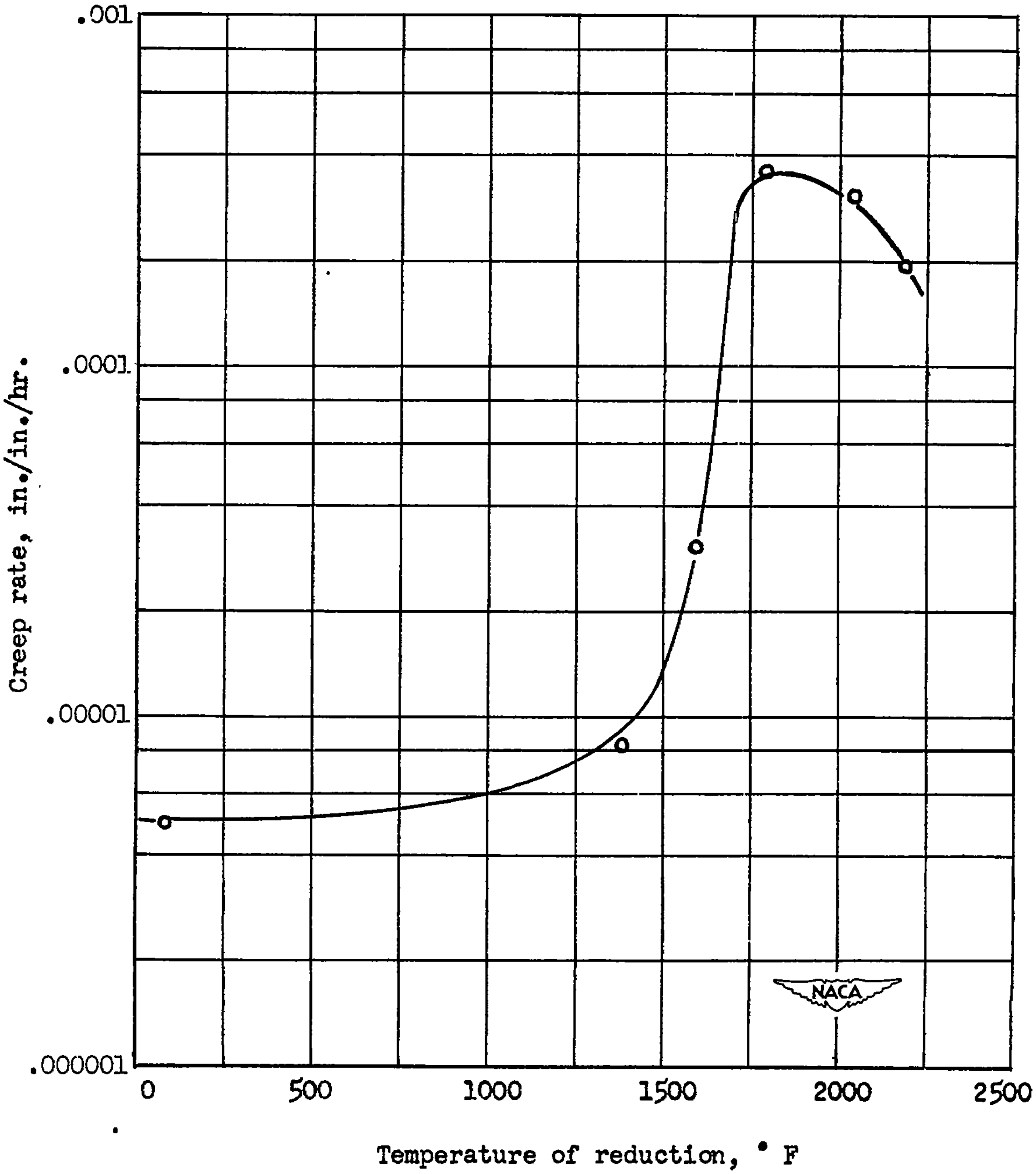


Figure 6.- Effect of rolling temperature on secondary creep rate under 50,000 psi at 1200° F of solution-treated low-carbon N-155 alloy reduced 15 percent.

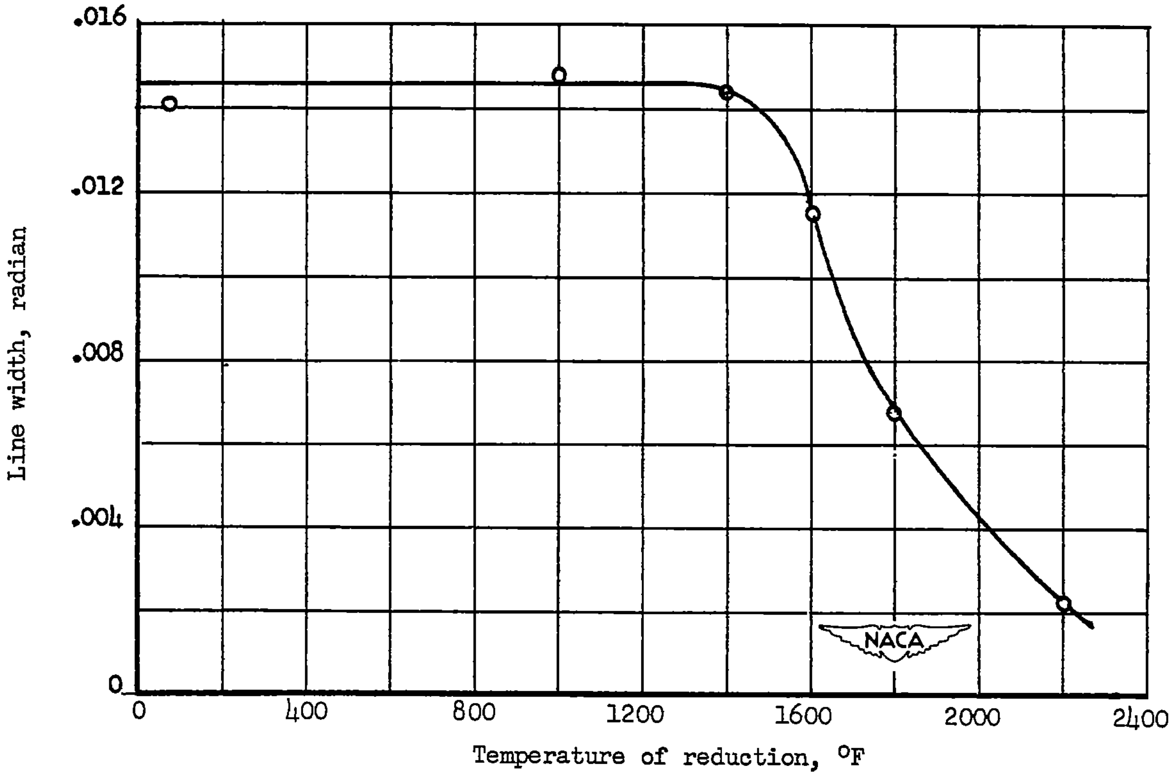


Figure 7.- Effect of rolling temperature on width of [220] line of solution-treated low-carbon N-155 alloy reduced 15 percent.

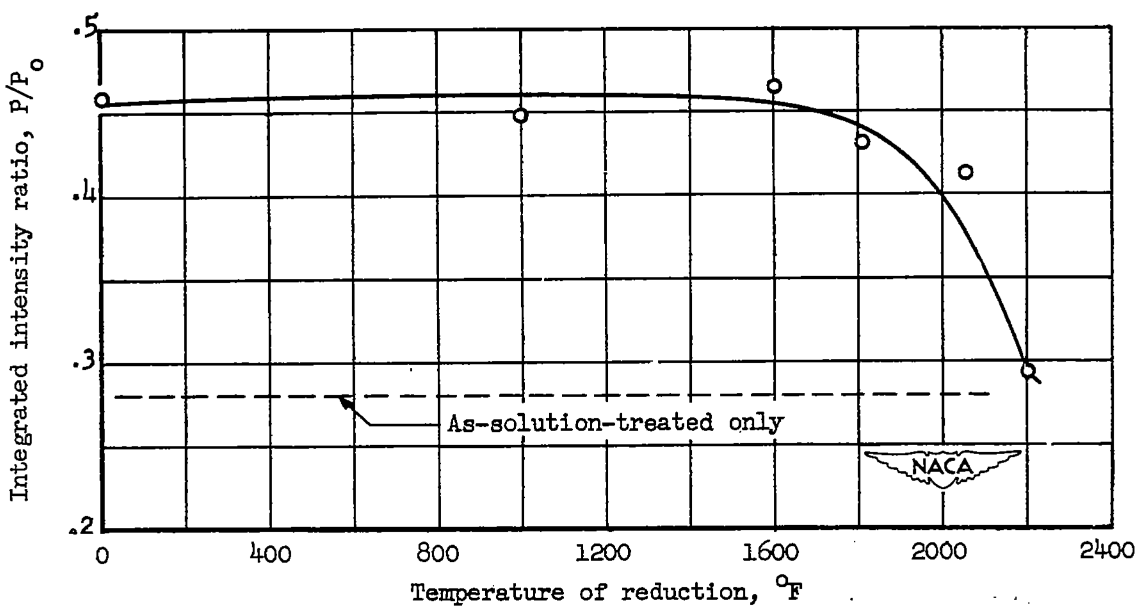
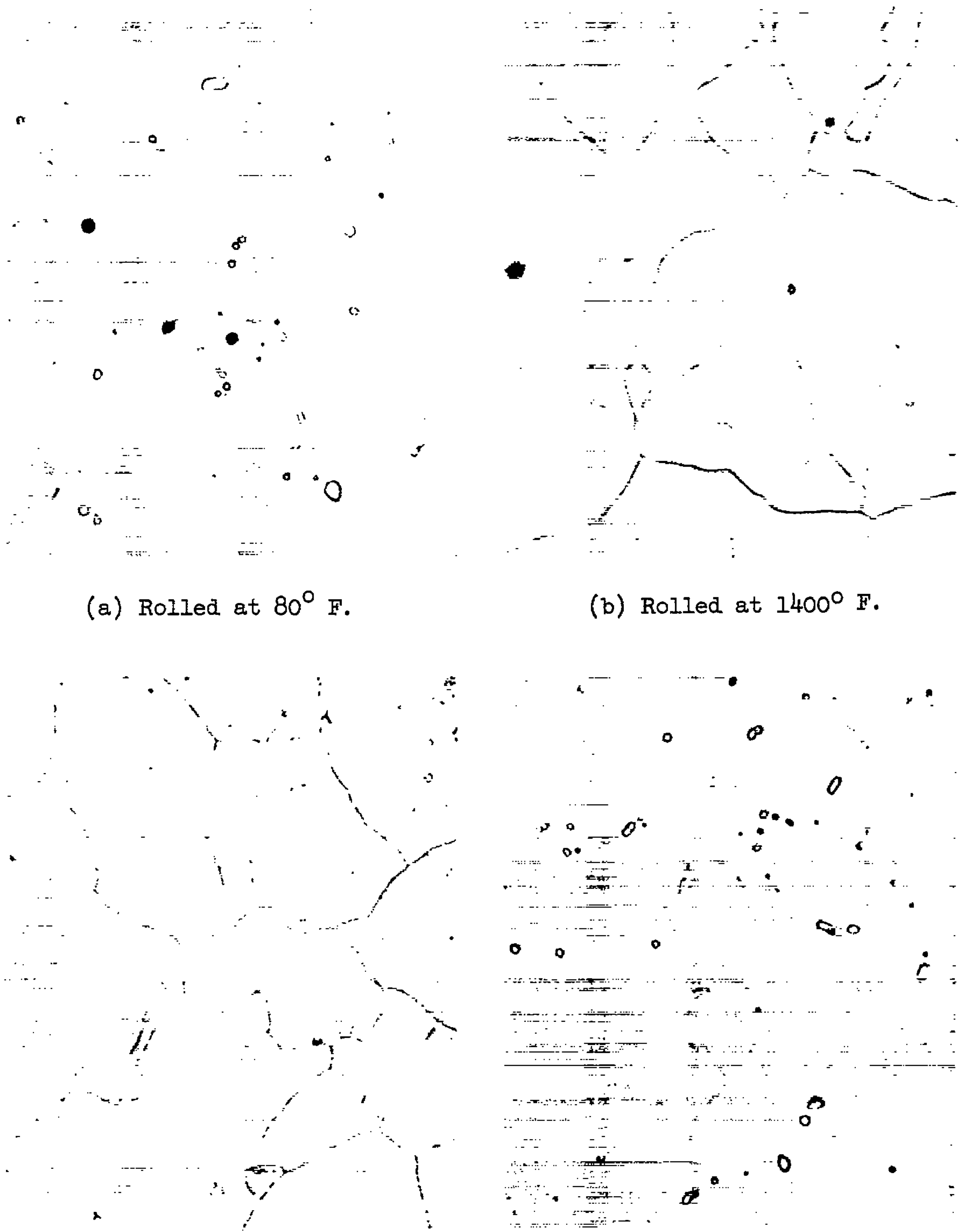


Figure 8.- Effect of rolling temperature on intensity of [111] line of solution-treated low-carbon N-155 reduced 15 percent. P, integrated intensity of [111] line of low-carbon N-155; P_o, integrated intensity of [111] line of nickel standard.



(a) Rolled at 80° F.

(b) Rolled at 1400° F.

(c) Rolled at 1800° F.

(d) Rolled at 2200° F.

Figure 9.- Effect of rolling temperature upon microstructure of solution-treated low-carbon N-155 bar stock reduced 15 percent. Electrolytically etched in chromic acid, X1000.

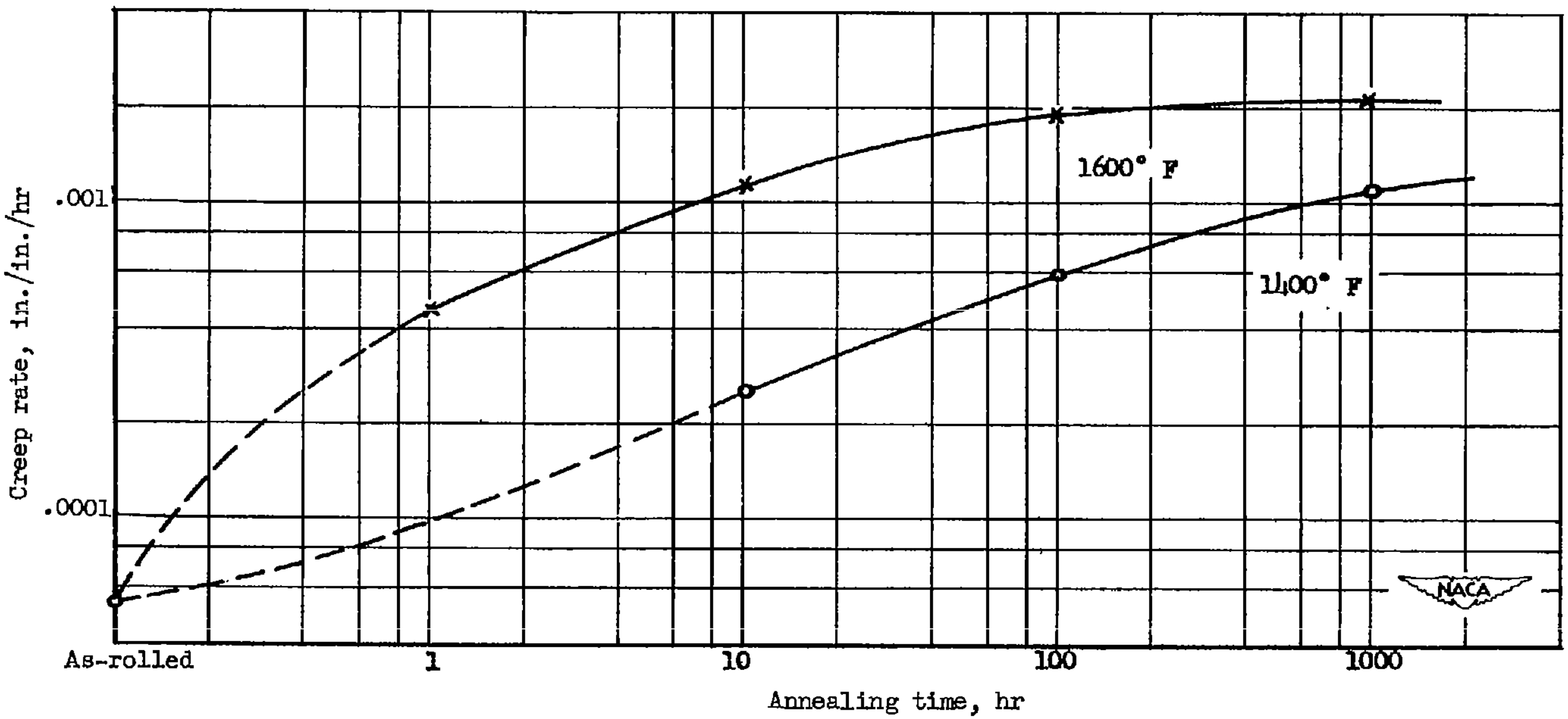


Figure 10.- Effect of annealing on secondary creep rate under 50,000 psi at 1200° F of solution-treated low-carbon N-155 alloy reduced 5 percent at 80° F.

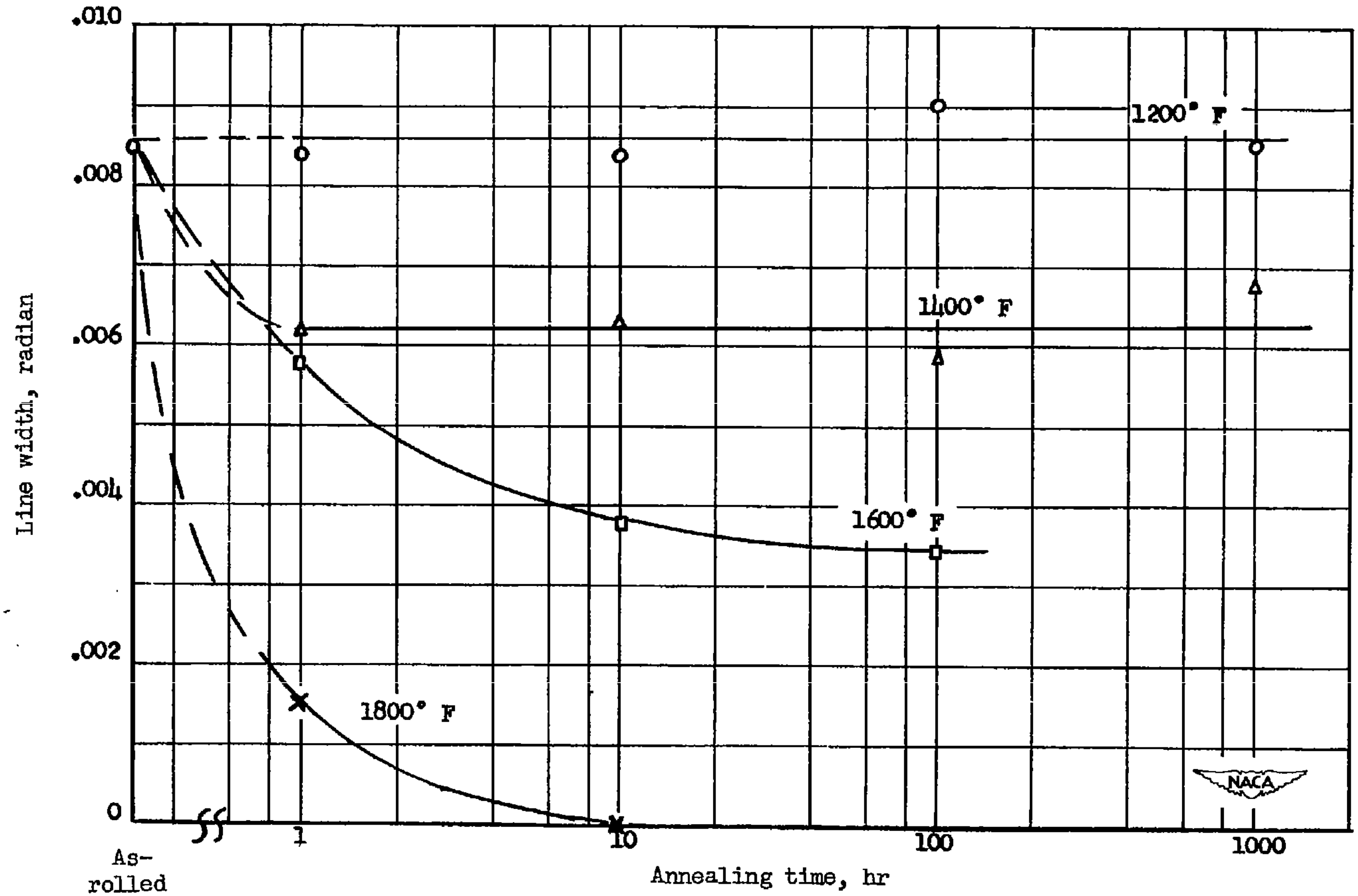


Figure 11.- Effect of annealing on width of [220] line of solution-treated low-carbon N-155 alloy reduced 5 percent at 80° F.

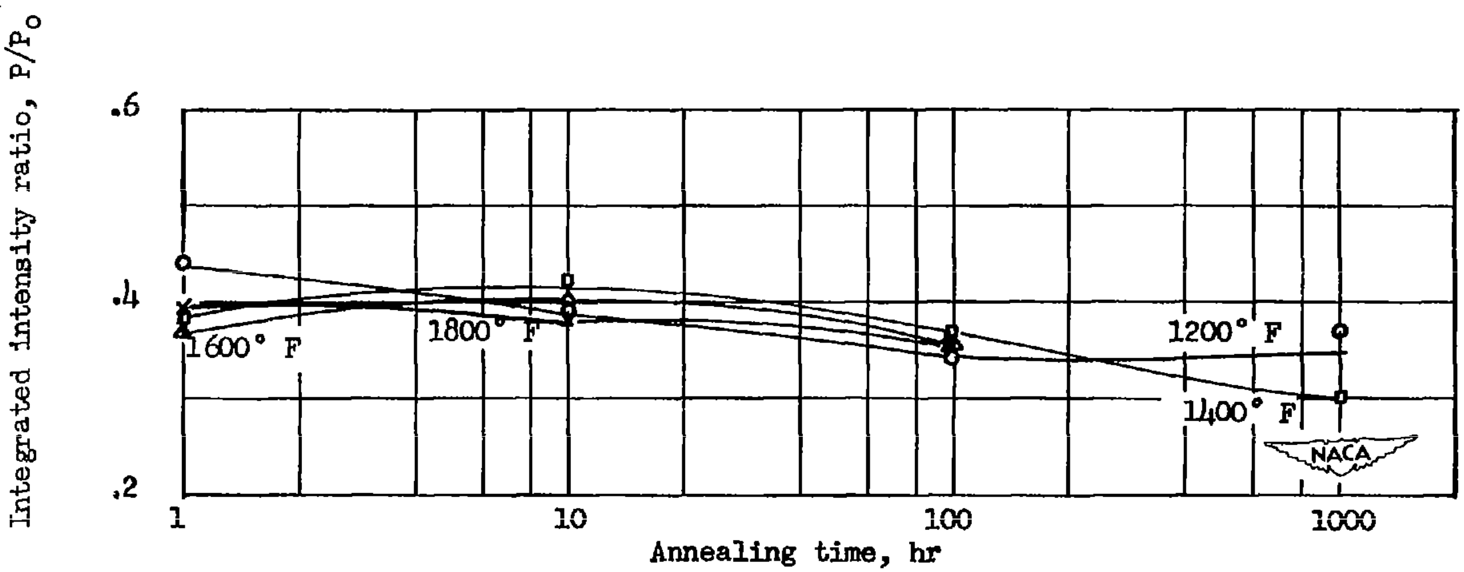


Figure 12.- Effect of annealing on [111]-line intensity of solution-treated low-carbon N-155 alloy reduced 5 percent at 80° F. P, integrated intensity of [111] line of low-carbon N-155; P_o, integrated intensity of [111] line of nickel standard.

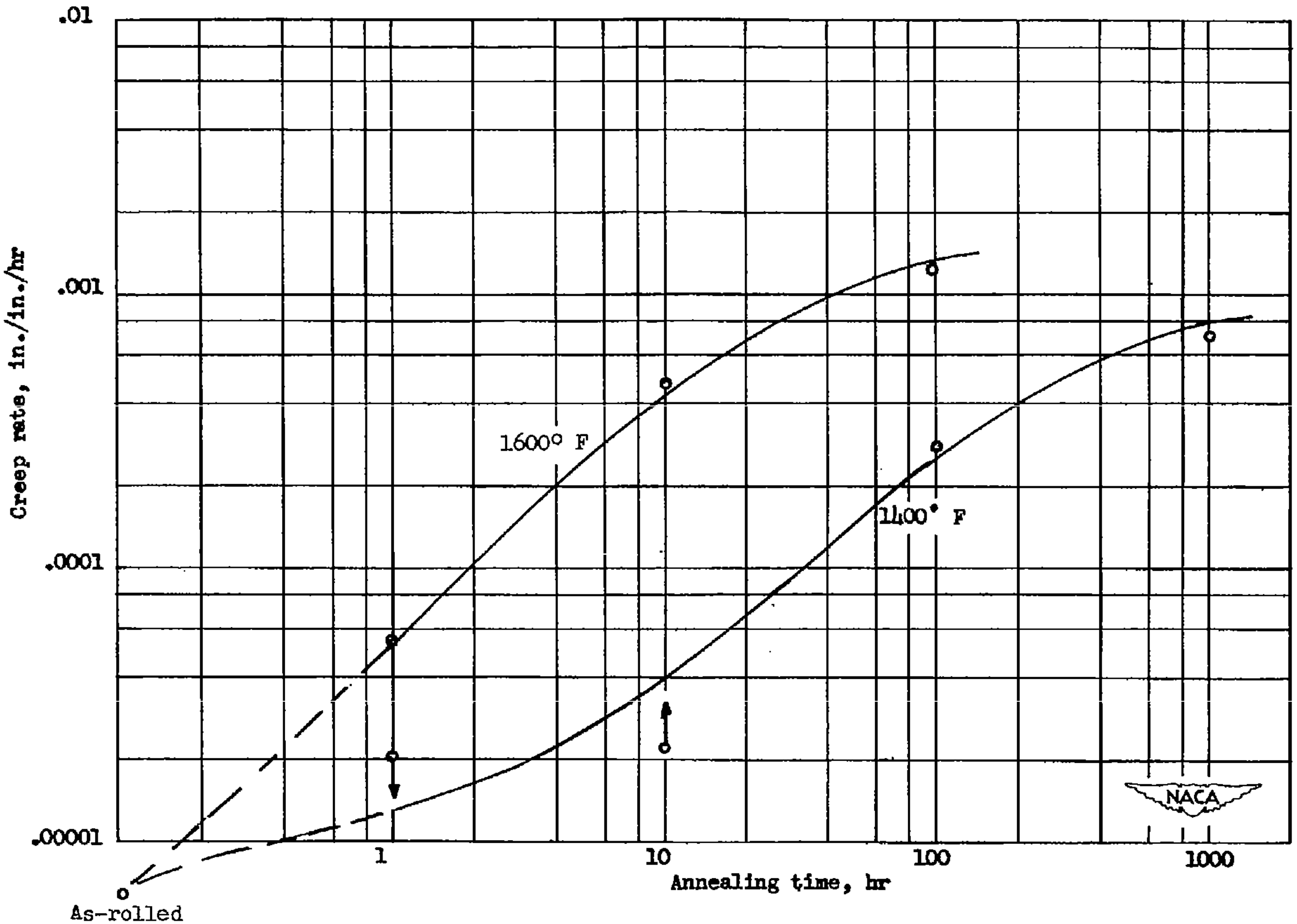


Figure 13.- Effect of annealing on secondary creep rate under 50,000 psi at 1200° F of solution-treated low-carbon N-155 alloy reduced 15 percent at 80° F.

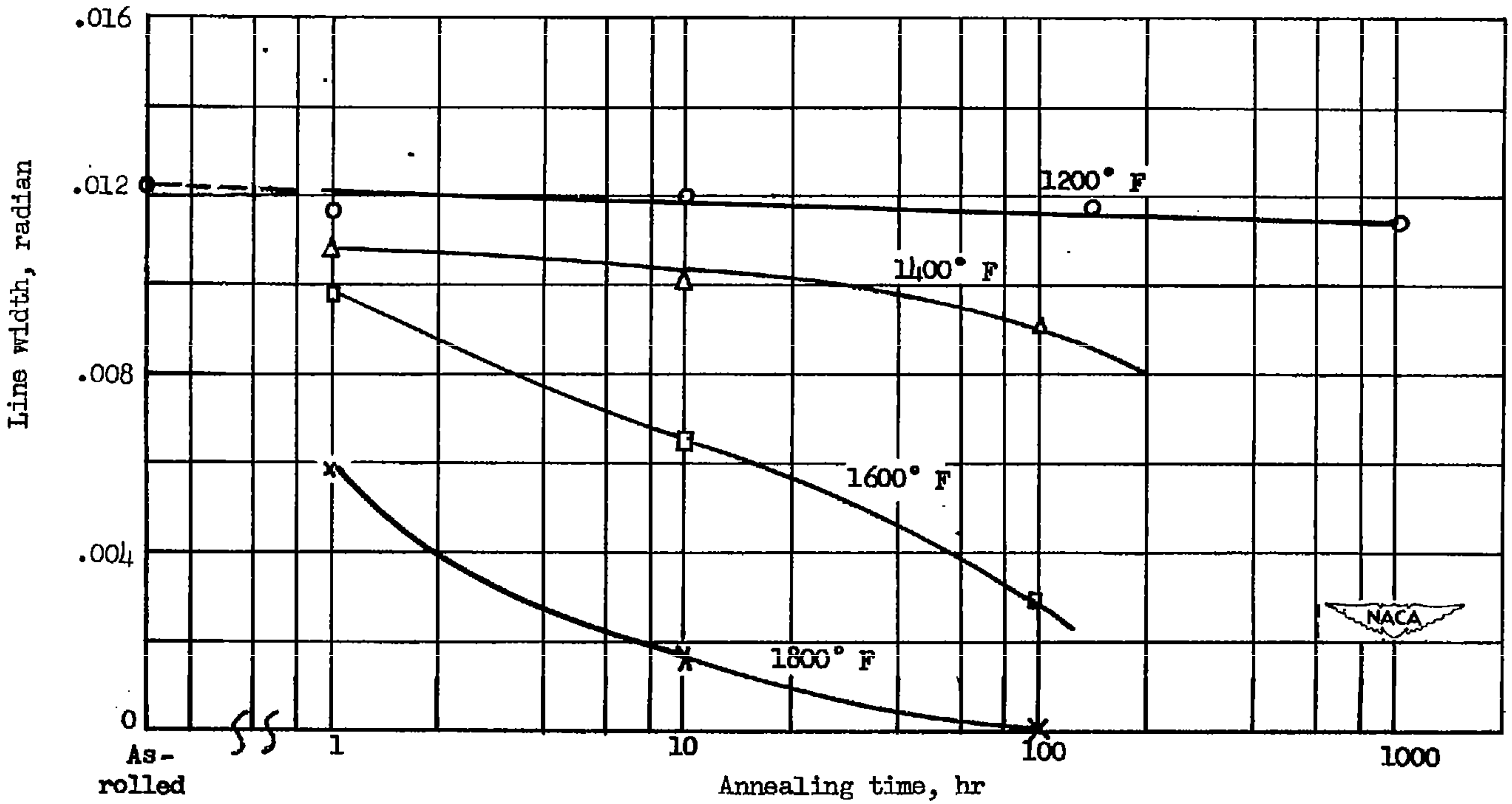


Figure 14.- Effect of annealing on width of [220] line of solution-treated low-carbon N-155 alloy reduced 13 percent at 80° F.

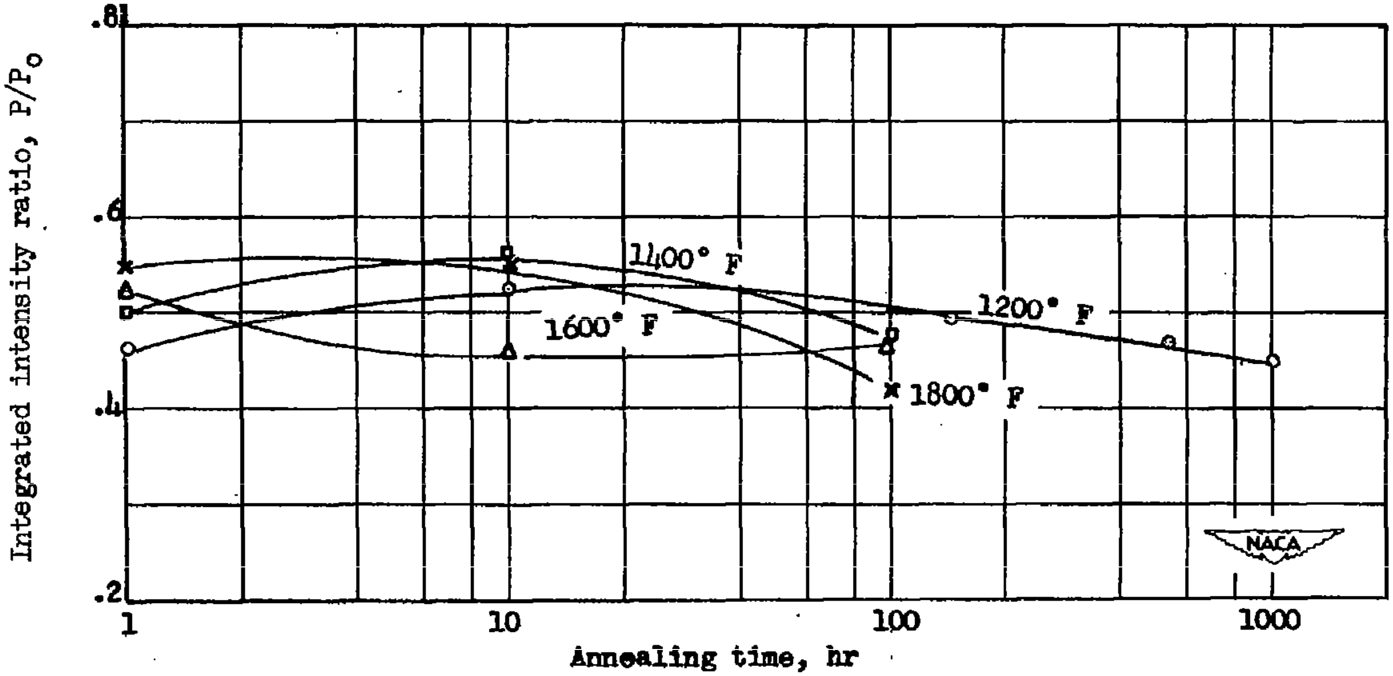


Figure 15.- Effect of annealing on [111]-line intensity of solution-treated low-carbon N-155 alloy reduced 15 percent at 80° F. P, integrated intensity of [111] line of low-carbon N-155; P_o, integrated intensity of [111] line of nickel standard.

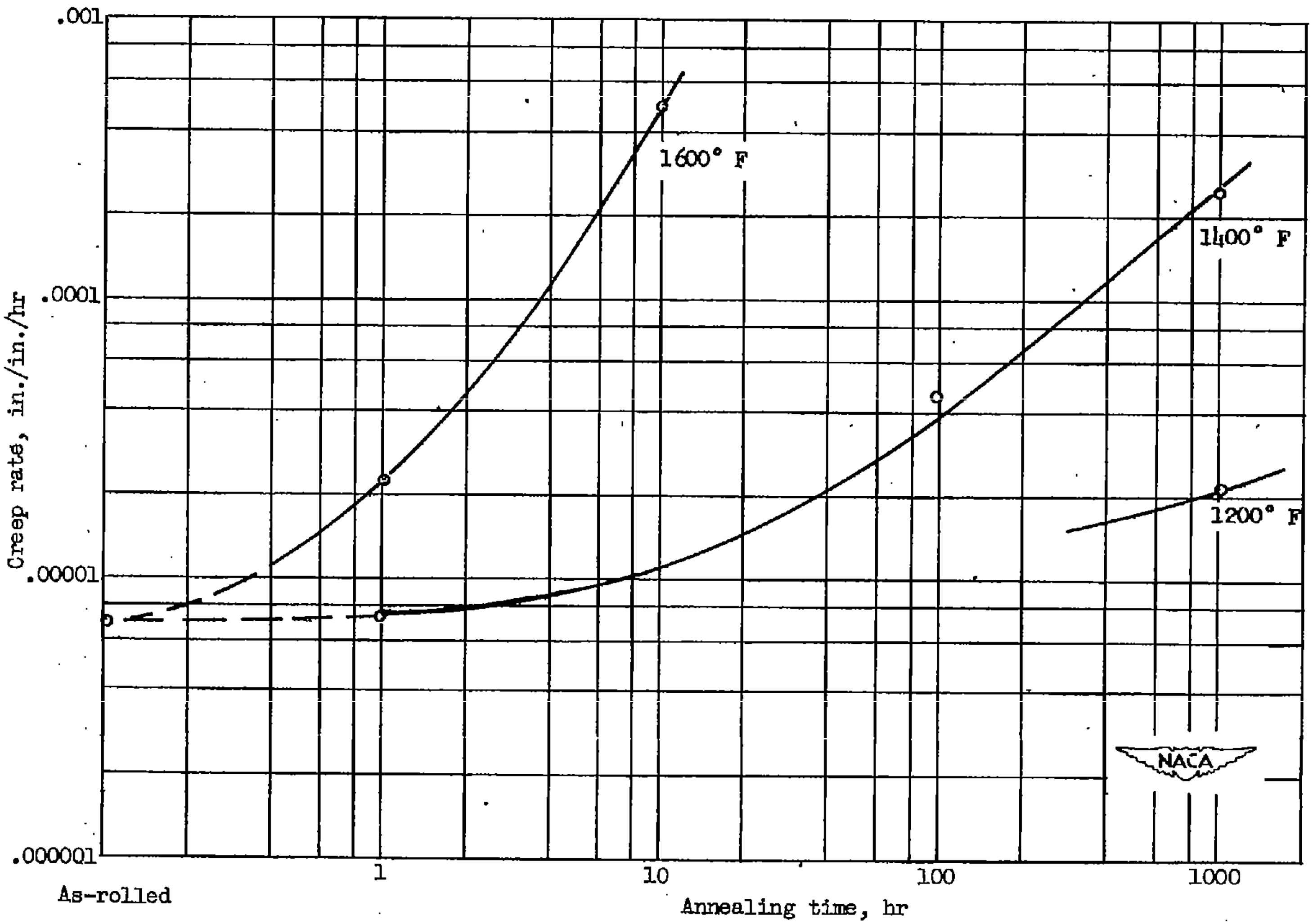


Figure 16.- Effect of annealing on secondary creep rate under 50,000 psi at 1200° F of solution-treated low-carbon N-155 alloy reduced 40 percent at 80° F.

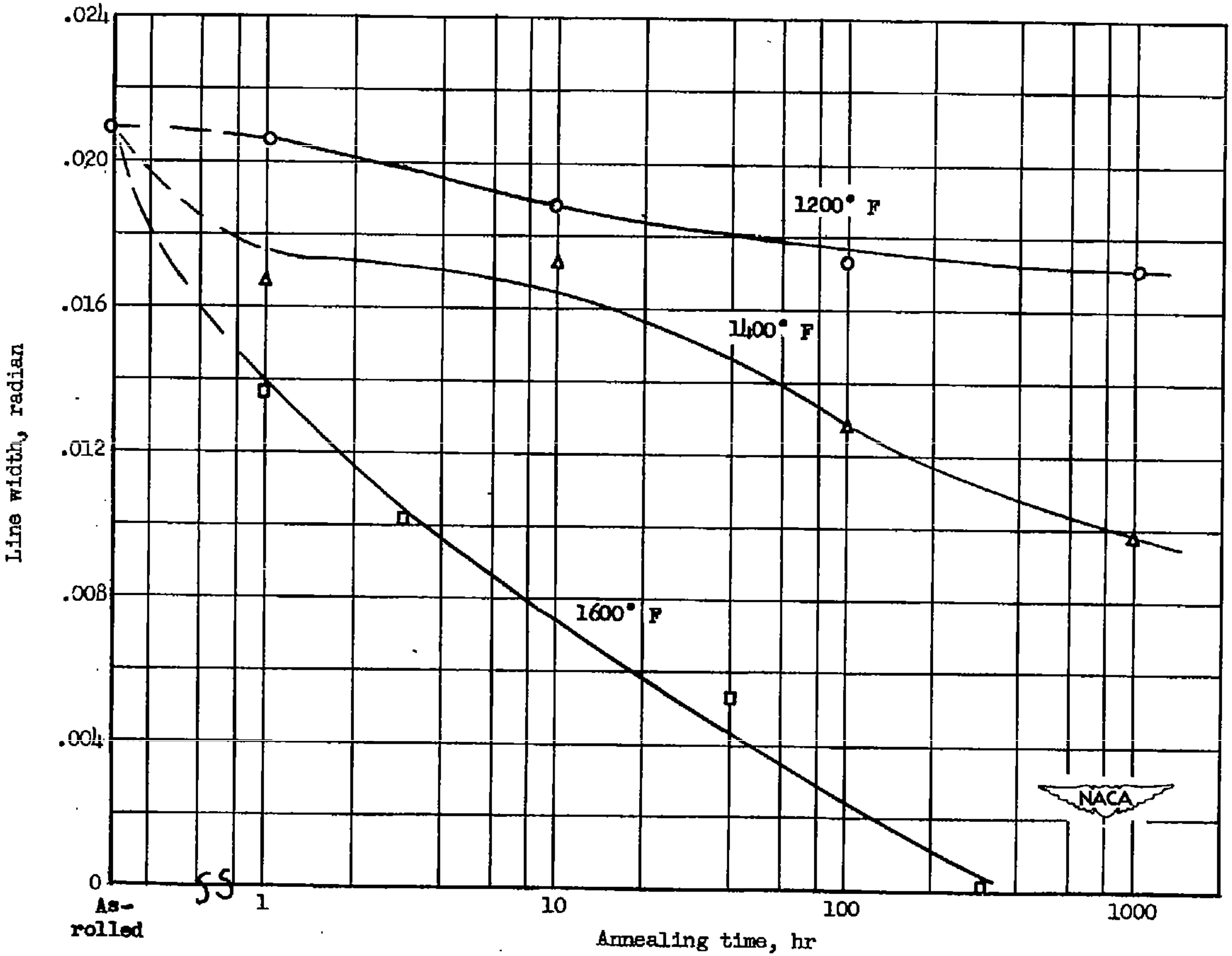


Figure 17.- Effect of annealing on width of [220] line of solution-treated low-carbon N-155 alloy reduced 40 percent at 80° F.

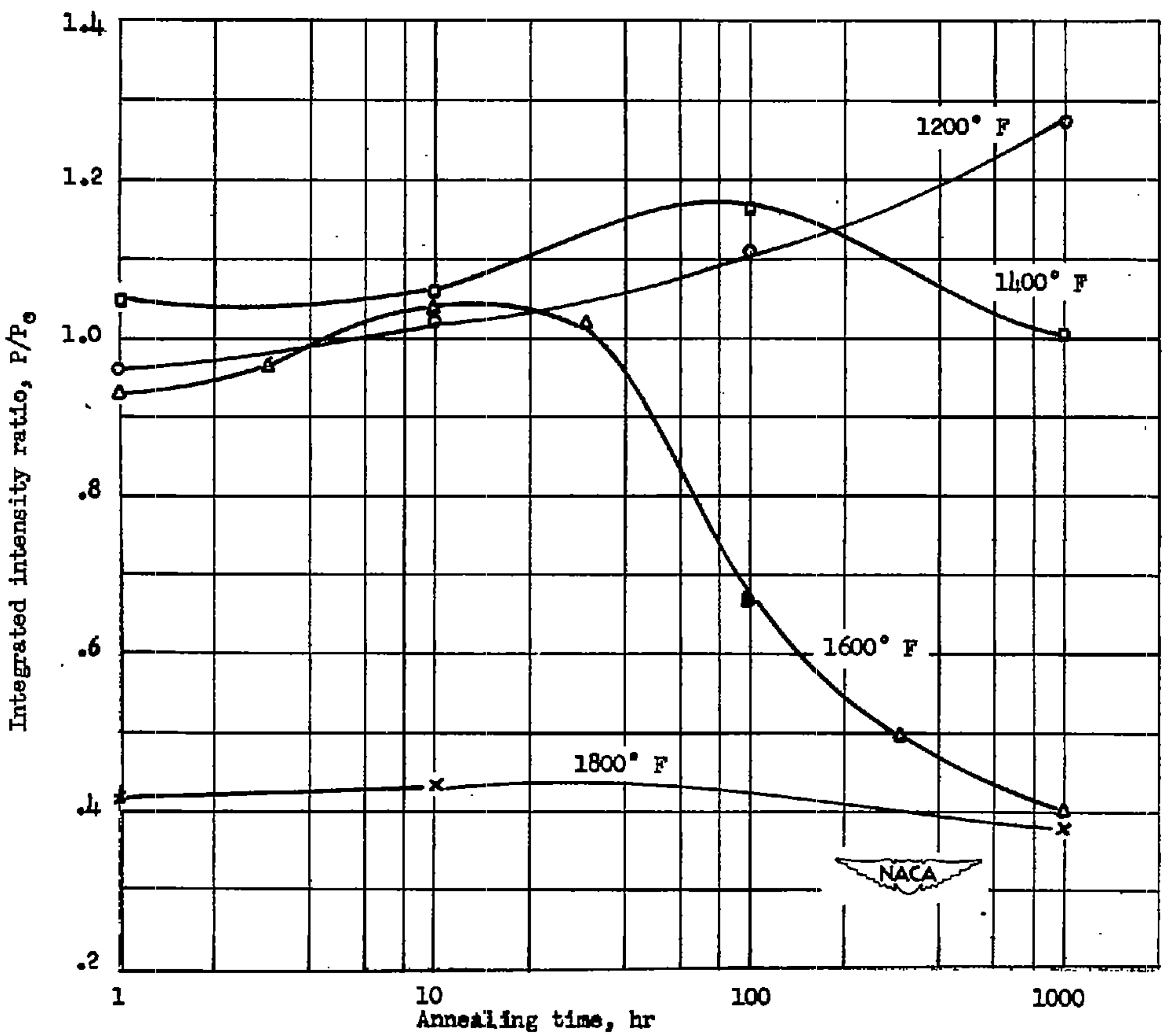


Figure 18.- Effect of annealing on integrated intensity of $\boxed{111}$-line of solution-treated low-carbon N-155 alloy reduced 40 percent at 80° F. P, integrated intensity of $\boxed{111}$ line of low-carbon N-155; P_o, integrated intensity of $\boxed{111}$ line of nickel standard.

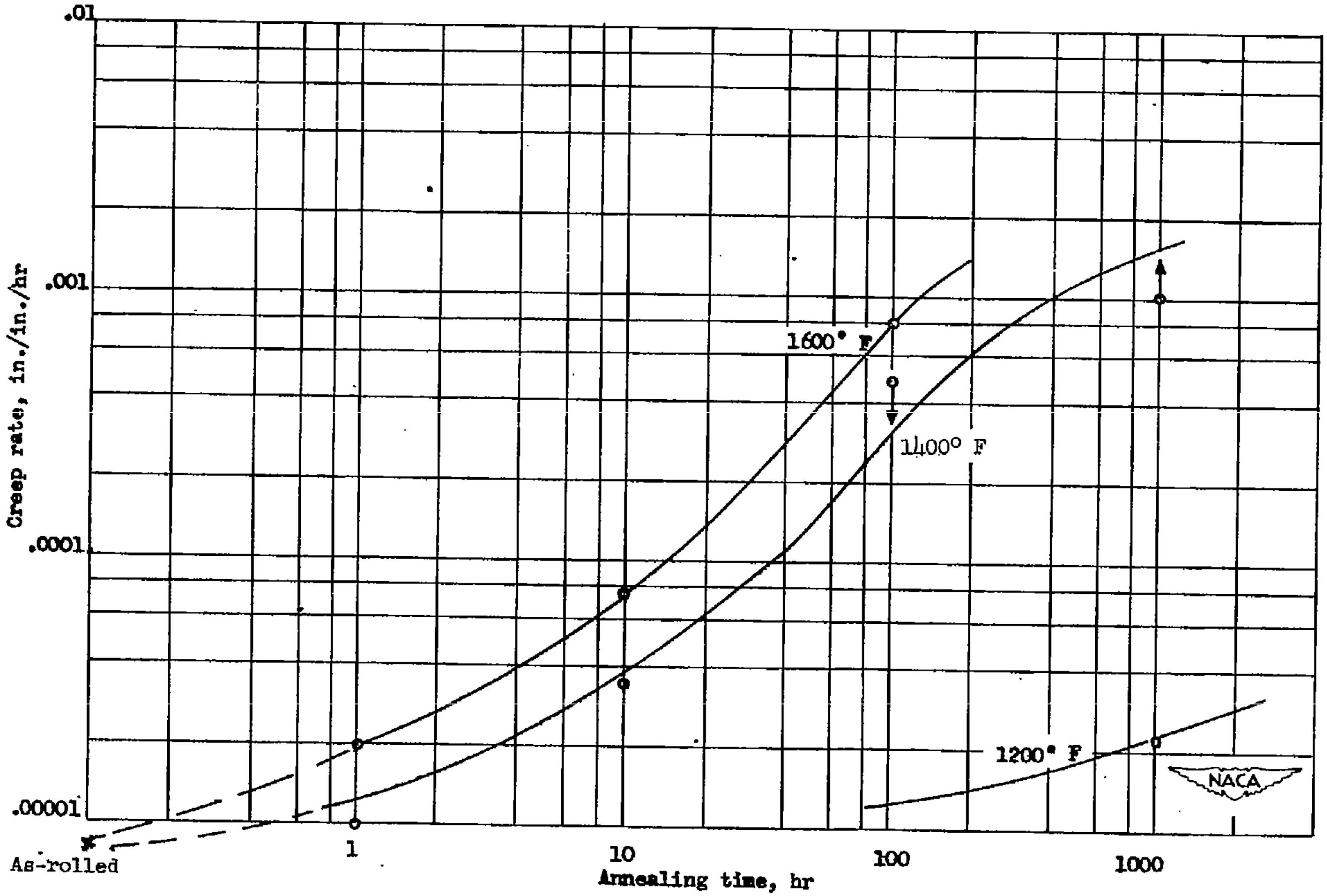


Figure 19.- Effect of annealing on secondary creep rate under 50,000 psi at 1200° F of solution-treated low-carbon N-155 alloy reduced 15 percent at 1400° F.

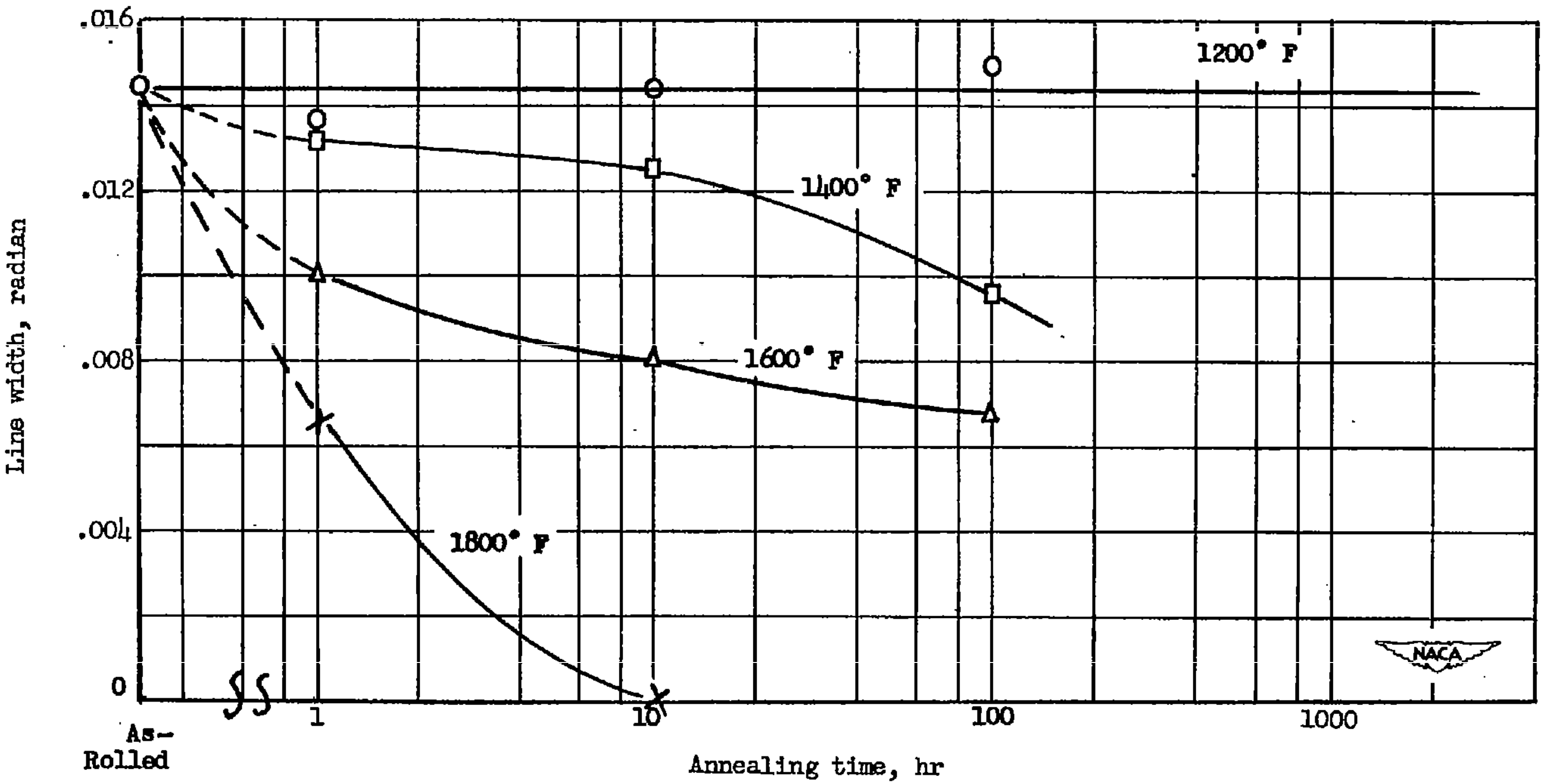


Figure 20.- Effect of annealing on width of [220] line of solution-treated low-carbon N-155 alloy reduced 15 percent at 1400° F.

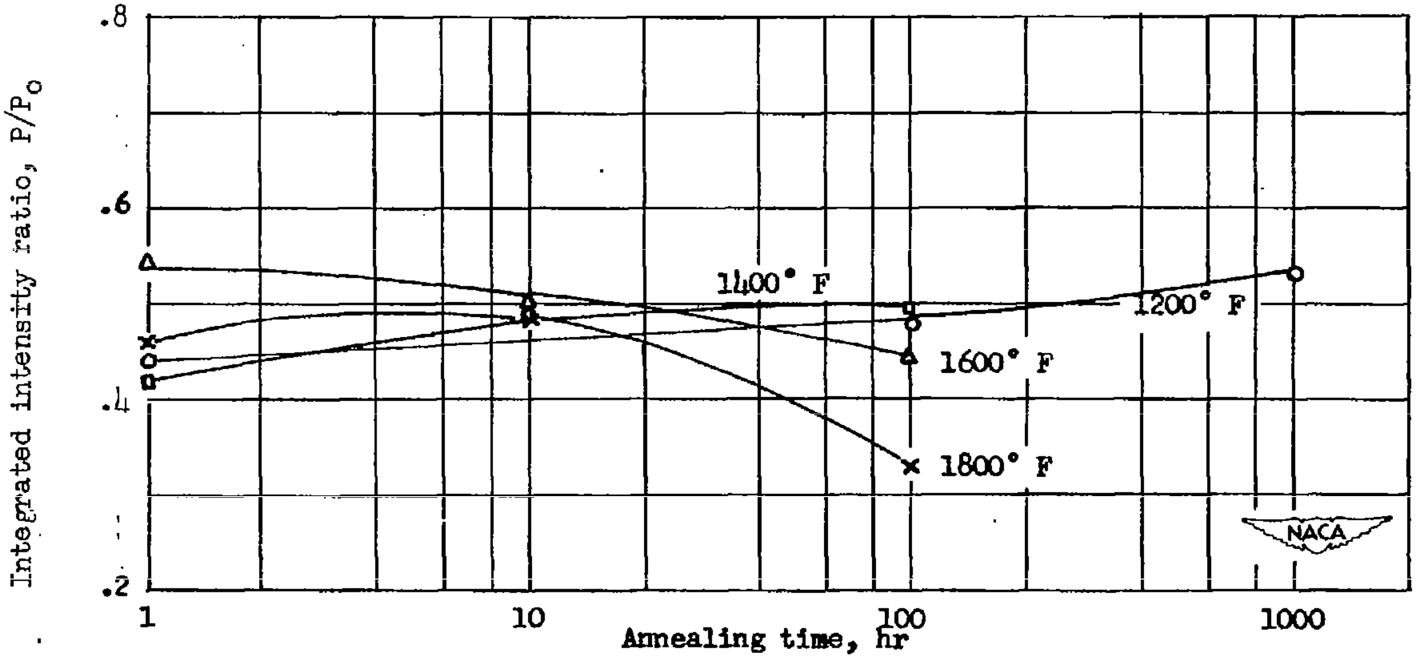
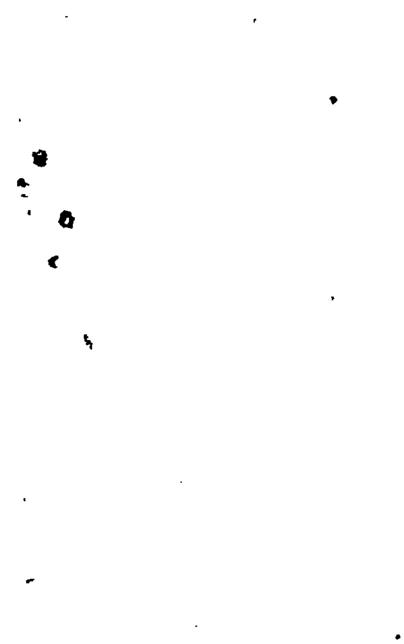


Figure 21.- Effect of annealing on [111]-line intensity of solution-treated low-carbon N-155 alloy reduced 15 percent at 1400° F. P, integrated intensity of [111] line of low-carbon N-155; P_o, integrated intensity of [111] line of nickel standard.

(a) Annealed 1 hour.

(b) Annealed 10 hours.

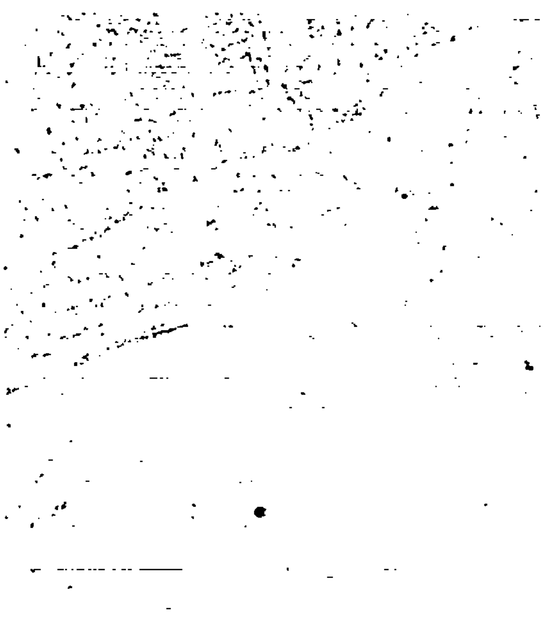


(c) Annealed 100 hours.

Figure 22.- Effect of annealing at 1800° F upon microstructure of solution-treated low-carbon N-155 bar stock reduced 15 percent at 1400° F. Electrolytically etched in chromic acid, X1000.

(a) Annealed 1 hour.

(b) Annealed 10 hours.



(c) Annealed 100 hours.

(d) Annealed 1000 hours.

Figure 23.- Effect of annealing at 1600° F upon microstructure of solution-treated low-carbon N-155 bar stock reduced 40 percent at 80° F. Electrolytically etched in chromic acid, X1000.

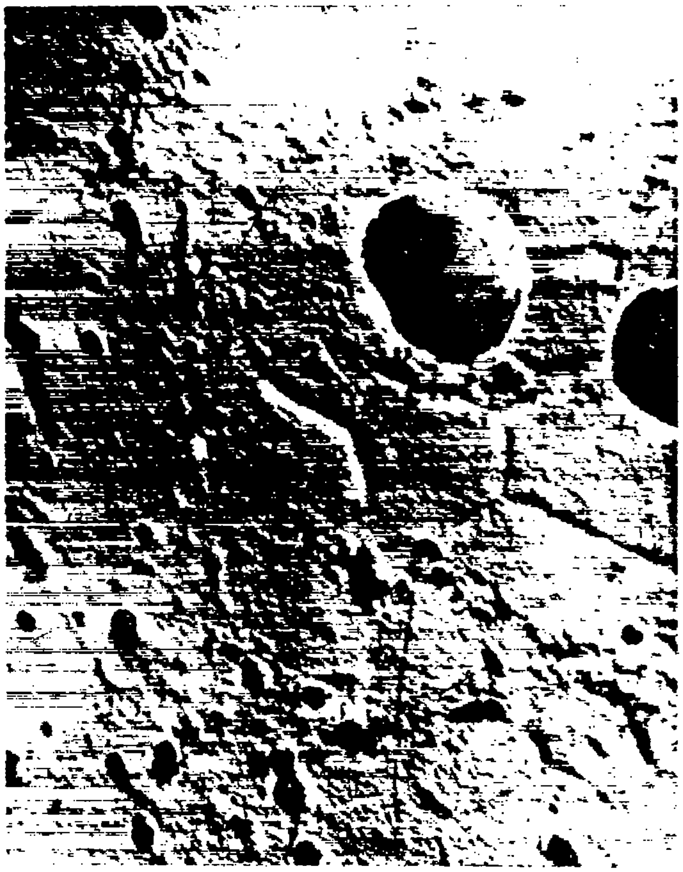


Figure 24.- Electron micrograph of solution-treated low-carbon N-155 bar stock reduced 40 percent at room temperature and annealed 10 hours at 1600° F, X10,000.

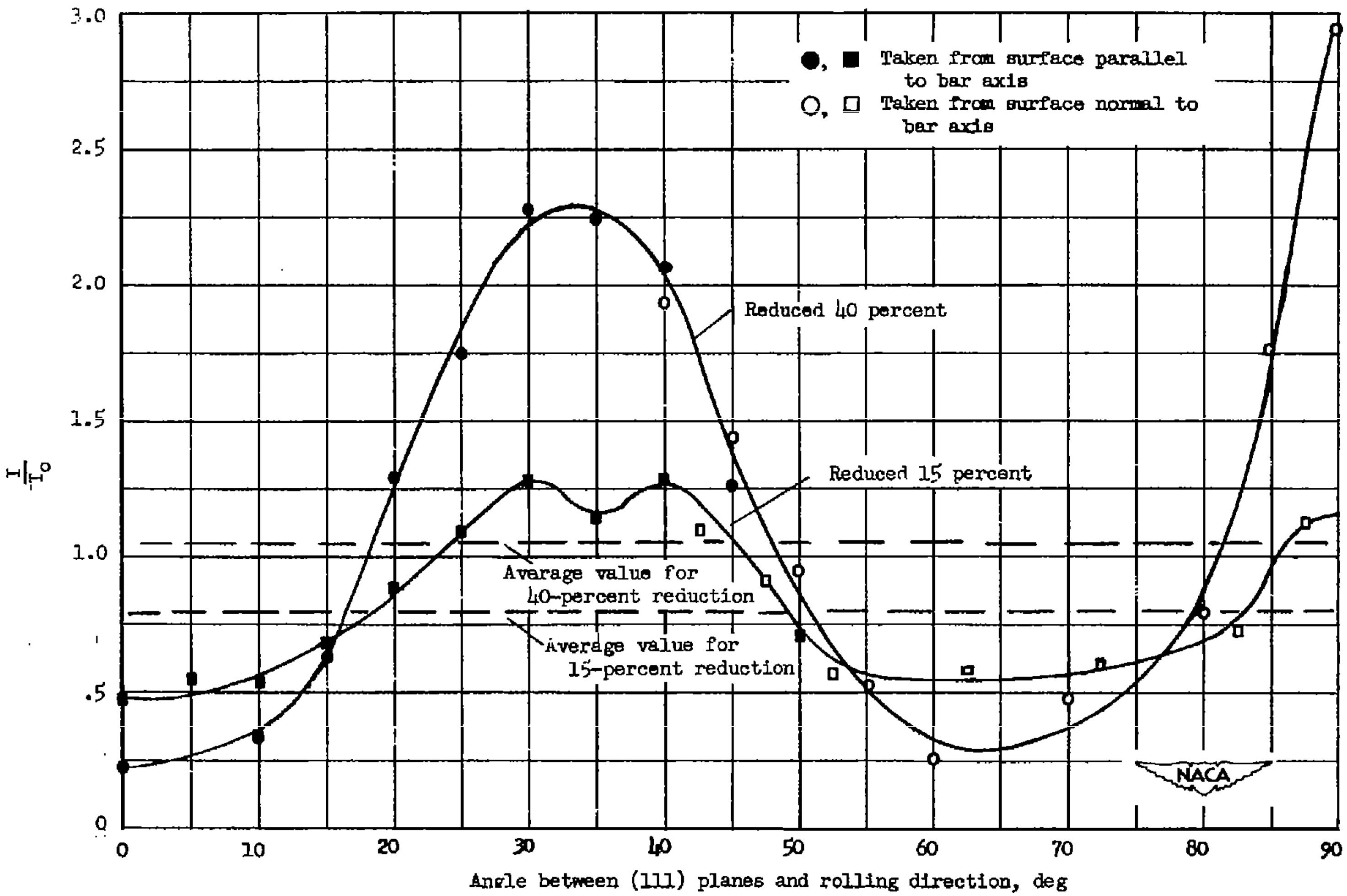


Figure 25.- Effect of rolling at 80° F on [111]-line intensity at indicated angles with the rolling direction of solution-treated low-carbon N-155 alloy. I, [111]-line intensity at indicated angle; I_o, [111]-line intensity of N-155 without extinction.

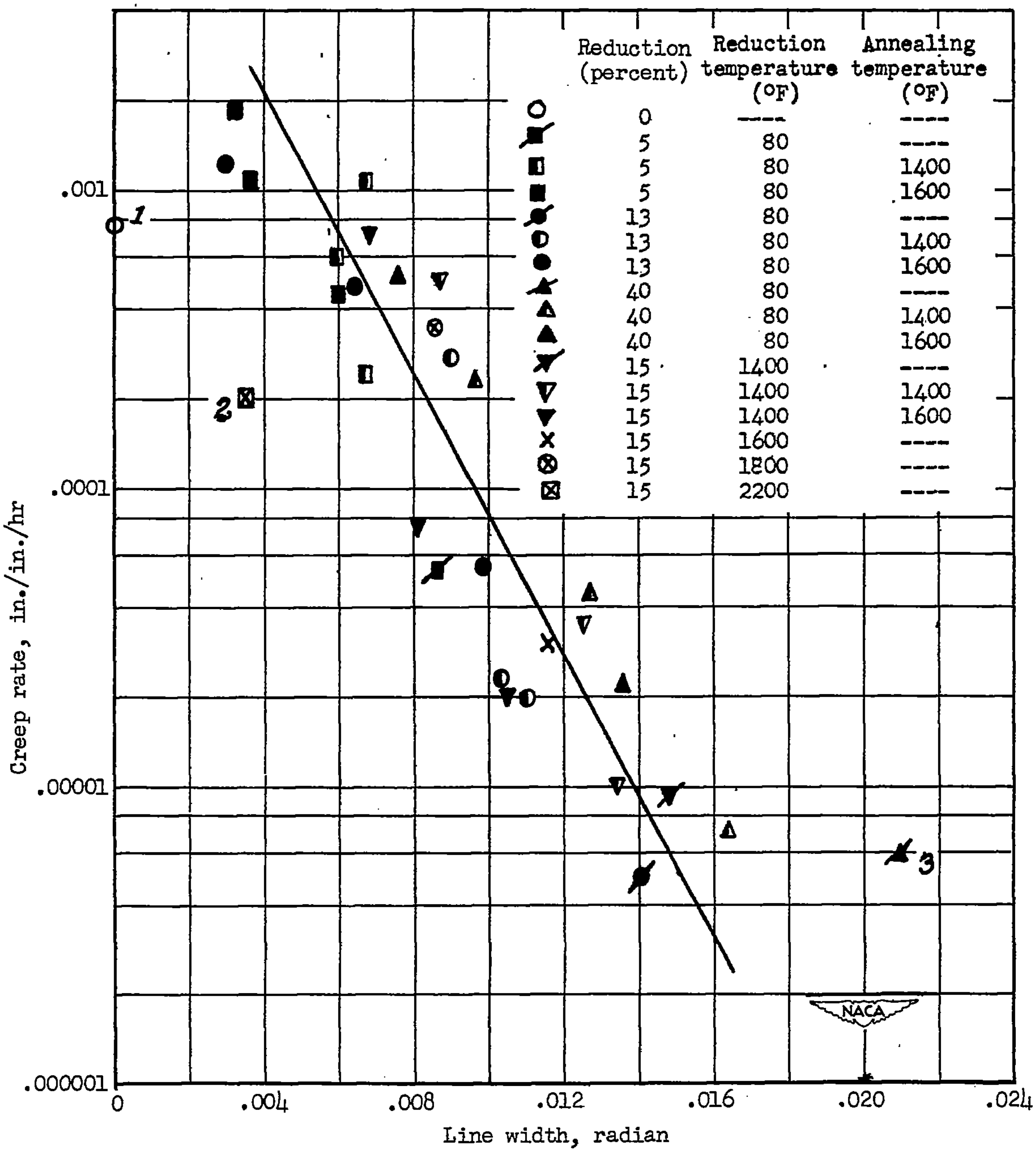


Figure 26.- Correlation of creep rate under 50,000 psi at 1200° F with [220]-line width of solution-treated, cold-worked, and annealed low-carbon N-155 alloy.

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